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DBs: USPAT; US-PGPUB

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☑ Highlight all hit terms initially

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1	☑	☐	☐	☐	US 20020112962 A1	20020822	10	Oxidising electrolytic method for obtaining a	205/102	204/230.6		Beauvir, Jacques	☐	☐	☐	☐	☐	☐
2	☑	☐	☐	☐	US 5201492 A	19930413		Metal diaphragm for diaphragm-type valve	251/331	92/103M		Beauvir, Jacques	☐	☐	☐	☐	☐	☐
3	☑	☐	☐	☐	US 4941504 A	19900717		Manual fluid-control valve with limited closing	137/524	137/556.3; 251/297;		Beauvir, Jacques	☐	☐	☐	☐	☐	☐

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1	☑	☐	☐	☐	US 20020170826 A1	20021121	7	Nickel electroforms	205/104	205/102		Wong, Kam Po et al.	☐	☐	☐	☐	☐	☐
2	☑	☐	☐	☐	US 6620303 B2	20030916		Process for making nickel electroforms	205/67	205/104		Wong, Kam Po et al.	☐	☐	☐	☐	☐	☐
3	☑	☐	☐	☐	US 6045681 A	20000404		Manufacturing method of planographic printing plate	205/214	204/DIG.9; 205/172;		Mori, Takahiro	☐	☐	☐	☐	☐	☐
4	☑	☐	☐	☐	US 5605615 A	19970225		Method and apparatus for plating metals	205/83	204/229.2; 204/229.7;		Goolsby, Peter G. et al.	☐	☐	☐	☐	☐	☐
5	☑	☐	☐	☐	US 5486280 A	19960123		Process for applying control variables having fractal	205/67	204/DIG.9; 205/102;		Bullock, IV, Jonathan S. et al.	☐	☐	☐	☐	☐	☐
6	☑	☐	☐	☐	US 5470673 A	19951128		Electrochromic and electrocatalytic material	429/44	205/241; 205/255;		Tseung, Alfred C. C. et al.	☐	☐	☐	☐	☐	☐
7	☑	☐	☐	☐	US 5141602 A	19920825		High-productivity method and apparatus for making	205/103	205/115; 427/142;		Chen, Chengjun J. et al.	☐	☐	☐	☐	☐	☐
8	☑	☐	☐	☐	US 4789437 A	19881206		Pulse electroplating process	205/76	204/DIG.9; 205/104;		Sing, Miu W. et al.	☐	☐	☐	☐	☐	☐
9	☑	☐	☐	☐	US 4666567 A	19870519		Automated alternating polarity pulse electrolytic	205/83	204/229.3; 204/DIG.9;		Loch, David M.	☐	☐	☐	☐	☐	☐
10	☑	☐	☐	☐	US 4540476 A	19850910		Procedure for making nickel electrodes	205/60	204/DIG.9; 429/223		Dyer, Christopher K.	☐	☐	☐	☐	☐	☐
11	☑	☐	☐	☐	US 4517059 A	19850514		Automated alternating polarity direct current	205/83	204/229.3; 204/DIG.9;		Loch, David M. et al.	☐	☐	☐	☐	☐	☐
12	☑	☐	☐	☐	US 4478689 A	19841023		Automated alternating polarity direct current	205/83	204/229.3; 204/DIG.9;		Loch, David M.	☐	☐	☐	☐	☐	☐

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2	US 6045681 A	25	☐	☑	☐	☐		USPAT
3	US 5605615 A	10	☐	☑	☐	☐		USPAT
4	US 5486280 A	7	☑	☐	☐	☐		USPAT
5	US 5470673 A	22	☑	☐	☐	☐		USPAT
6	US 5141602 A	27	☑	☐	☐	☐		USPAT
7	US 4789437 A	14	☑	☐	☐	☐		USPAT

US-PAT-NO: 5605615

DOCUMENT-IDENTIFIER: US 5605615 A

TITLE: Method and apparatus for plating metals

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Detailed Description Text - DETX (5):
A plating cell driver circuit (driver circuit) 30 is coupled to an input 20 and electrode 12. Plating cell driver circuit 30 receives a control signal from a waveform generator 20. Control signals may be generated by a waveform generator source or other means. In voltage forcing mode, plating cell driver circuit 30 alters either the applied voltage pulse characteristics with regard to the multiple voltage levels and duty cycles that are applied to plating cell 10 in response from software algorithms controlled by computer system 400 that stimulate an electrochemical reaction, producing the desired metallic deposit characteristics in plating cell 10. In current forcing mode, plating cell driver circuit 30 alters the applied current pulse characteristics with regard to the multiple current levels and duty cycles that are applied to plating cell 10 in response from software algorithms controlled by computer system 400 that stimulate an electrochemical reaction, producing the desired metallic deposit characteristics in plating cell 10.

Detailed Description Text - DETX (6):
Switch 60 selects the mode of operation for the system. In the default position, switch 60 selects voltage forcing mode, whereby driver 30 outputs a programmable pulse train of multiple levels of voltages at programmable duty cycles and programmable frequencies. Switch 60 is a sense point selector that is controlled by a digital signal source from a computerized control element 75. Switch 60 routes the voltage sensed from electrode 12 through a feedback network of wires to the input of driver 30 and waveform generator 20. The sensed voltage at electrode 12 provides a means to close a feedback loop to driver circuit 30 and waveform generator 20 to ensure stability and compliance to programmed voltages from small signal waveform generator 20 through amplifier 30, and at the electrode 12.

Detailed Description Text - DETX (7):
In the secondary position, switch 60 selects current forcing mode, whereby driver 30 outputs a programmable pulse train of multiple levels of currents at programmable duty cycles and programmable frequencies. Switch 60 routes the current sensed through plating cell 10 to the input of driver circuit 30 and waveform generator 20. The sensed current through plating cell 10 provides a means to close a feedback loop to driver circuit 30 and waveform generator 20 to ensure amplifier stability and compliance to programmed currents from small signal waveform generator 20 through driver circuit 30, and through plating cell 10. The operation of the plating system in current sensing mode and voltage sensing mode will be further described below.

Detailed Description Text - DETX (12):
Frequency detector 92 and digital counter 93 are both coupled to software 94. Software 94 determines, displays on a video display 95, and tracks the

United States Patent [19]

Goolsby et al.

[11] Patent Number: 5,605,615

[45] Date of Patent: Feb. 25, 1997

[54] METHOD AND APPARATUS FOR PLATING METALS

[75] Inventors: Peter G. Goolsby, Phoenix; Dan R. Ramirez, Chandler; Lei P. Lai, Glendale, all of Ariz.

[73] Assignee: Motorola, Inc., Schaumburg, Ill.

[21] Appl. No.: 349,590

[22] Filed: Dec. 5, 1994

[51] Int. Cl.[6] C25D 21/12; C25D 5/18; C25D 5/50; G01N 27/26

[52] U.S. Cl. 205/83; 205/103; 205/104; 205/105; 205/106; 205/107; 205/108; 204/406; 204/434; 204/400; 204/DIG. 9; 204/228

[58] Field of Search 204/406, 434, 204/400, 228; 205/83, 103, 104, 105, 107, 108, 102, 106

[56] References Cited

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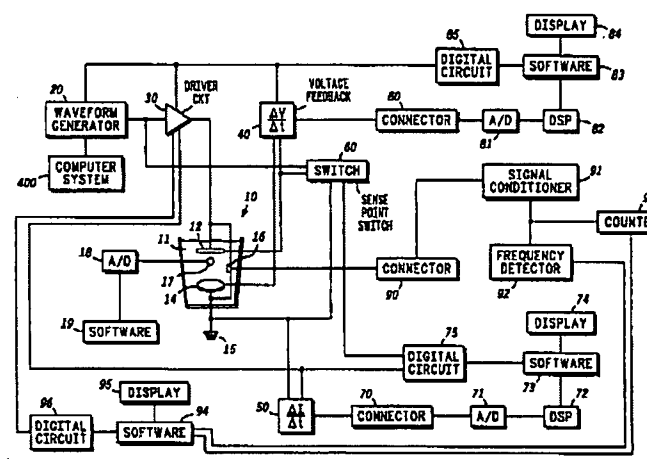
Primary Examiner—Kathryn Gorgos
Assistant Examiner—Edna Wong
Attorney, Agent, or Firm—Miriam Jackson

[57] ABSTRACT

A method and apparatus for plating metals which delivers a voltage pulse with the possibility of a widely varying current magnitude characteristic to a plating electrode and an object having a large electrical reactance in terms of a parallel resistance and capacitance in order to raise the voltage potential between the electrode and an object to a programmed plating voltage overpotential and underpotential. The programmed plating voltage overpotential determines how fast the electrochemical reaction is allowed to proceed in the diffusion layer, and the programmed voltage underpotential determines how quickly the electrochemical reaction of the diffusion layer will slow down.

12 Claims, 3 Drawing Sheets



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5	US 5470673 A	22	☑					USPAT
6	US 5141602 A	27	☑					USPAT
7	US 4789437 A	14	☑					USPAT

US-PAT-NO: 5486280

DOCUMENT-IDENTIFIER: US 5486280 A

TITLE: Process for applying control variables having fractal structures

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Detailed Description Text - DETX (4):

With reference to FIG. 1, electroforming is achieved by placing a substrate 10 within an electrolytic solution 12. Current is passed between the substrate 10 and the electrode 14 to cause the deposition of components of the electrolyte on the substrate 10. The electrode 14 is connected to the galvanostat 16 by a counter electrode connection 17, while the substrate 10 is connected to the galvanostat 16 by a working electrode connection 19. The waveform of the current applied by the galvanostat 16 is controlled by a waveform generator 20 which is in turn controlled by a microprocessor 18, loaded with appropriate software. The power required by the galvanostat is obtained from the power source 21, which for example can be 120 VAC mains.

Detailed Description Text - DETX (6):

The fractal waveform is designed and, on command, is impressed as an input signal on a power amplifier, such as a galvanostat, such that a plating process can be driven by the power amplifier in a manner described herein. This is achieved by generating the design of the fractal waveform using a software program running on a personal computer, with output of the waveform to an arbitrary waveform generator, or by using a program located on a PROM chip which is housed in a waveform generator dedicated to electroforming in accordance with the present invention. In either case, the self-similar design of the waveform permits complete specification of a complex waveform with a relatively small number of parameters, each one being not arbitrary, but related to real physical properties of the plating system, especially mass-transfer characteristics.

Detailed Description Text - DETX (14):

The specific legend items are defined as follows: t.sub.0 is equal to the smallest "clock" (time) interval and is included in the starting position at zero current to cope with an artifact of waveform generator operation; t.sub.1 is the length of the rectangular cathodic pulse, typically equal to t.sub.0 and for most of the experiments performed was equal to 1 millisecond; t.sub.2 is the time at zero current following the cathodic pulse, and is typically 4-9 times t.sub.1 ; t.sub.1 and t.sub.2 taken together constitute the "cathodic pulse," which is repeated n.sub.1 times; i.sub.c is the amplitude of the cathodic pulse measured in unites of current; i.sub.a is the maximum amplitude of the anodic pulse in current units, is constrained to certain current density values, and as a first approximation will be the same at all scales; C.sub.c is the number of coulombs per cathodic pulse and for a rectangular pulse is equal to the product of i.sub.c times t.sub.1 ; and C.sub.A1 is the number of coulombs per anodic pulse associated with the shortest repeating period, while C.sub.A2, C.sub.A3, etc. are associated with successively longer periods. After a train of n cathodic pulses, each of level i and length t, which accumulate n*i*t coulombs, an anodic pulse carrying f*n*i*t coulombs is passed.

United States Patent [19]

Bullock, IV et al.

[11] **Patent Number: 5,486,280**

[45] **Date of Patent: Jan. 23, 1996**

[54] **PROCESS FOR APPLYING CONTROL VARIABLES HAVING FRACTAL STRUCTURES**

[75] Inventors: Jonathan S. Bullock, IV, Oak Ridge; Roger L. Lawson, Oliver Springs, both of Tenn.

[73] Assignee: Martin Marietta Energy Systems, Inc., Oak Ridge, Tenn.

[21] Appl. No.: 326,425

[22] Filed: Oct. 20, 1994

[51] Int. Cl.[6] C25D 1/00

[52] U.S. Cl. 205/67; 205/102; 205/103; 205/104; 204/DIG. 9

[58] Field of Search 205/87, 102, 103, 205/104; 204/DIG. 9, 228

[56] References Cited

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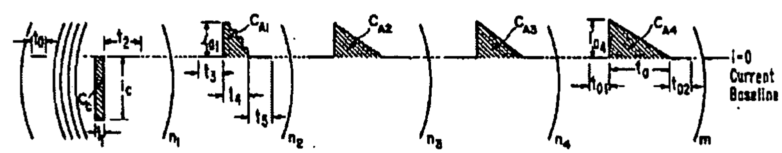
Primary Examiner—John Niebling
Assistant Examiner—Brendan Mee
Attorney, Agent, or Firm—E. A. Pennington; V. A. Branton; H. W. Adams

[57] ABSTRACT

A process and apparatus for the application of a control variable having a fractal structure to a body or process. The process of the present invention comprises the steps of generating a control variable having a fractal structure and applying the control variable to a body or process reacting in accordance with the control variable. The process is applicable to electroforming where first, second and successive pulsed-currents are applied to cause the deposition of material onto a substrate, such that the first pulsed-current, the second pulsed-current, and successive pulsed currents form a fractal pulsed-current waveform.

17 Claims, 2 Drawing Sheets



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6	US 5141602 A	27	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
7	US 4789437 A	14	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

US-PAT-NO: 5141602

DOCUMENT-IDENTIFIER: US 5141602 A

TITLE: High-productivity method and apparatus for making customized interconnections

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Detailed Description Text - DETX (39):
Electronic controller 88 comprises a microcomputer 101 with a data acquisition board 102, which controls an ac waveform generator 103, and an electrically controlled constant voltage or constant current amplifier 104. An ac ammeter 105 and an ac voltmeter 106, measures the voltage and the current on line 99.

Detailed Description Text - DETX (40):
Acquisition board 102 couples a digital output signal indicative of frequency from digital output device 111 over lead 108 to an input of ac waveform generator 103. Acquisition board 102 couples a digital output signal indicative of amplifier mode (voltage or current) from digital output device 111 over lead 109 to an input of Ac voltage/current power amplifier.

Detailed Description Text - DETX (44):
In FIGS. 9A and 9B like references are used for functions corresponding to the apparatus of FIG. 8. The crystal oscillator 123, divide by four frequency divider 124 and programmable frequency divider 125 provide a stable source of frequencies between 1 KHz and 64 KHz. The frequency can be set by either the computer controller 121 or by switches. The voltage reference 131 accepts a voltage provided over lead 112 by the controller 121 or can generate a voltage set locally that varies between 1 mV and 10 V. The unity gain inverting amplifier 132 takes the reference voltage and generates a negative voltage of the same magnitude. The waveform generator 136 creates a square wave whose peak amplitude is equal to the output of the voltage reference 131 and whose frequency is set by the output of the programmable frequency divider 125. The square wave has an average value of 0 Volts. The voltage/current mode select circuit 137 determines whether the self induced repair is occurring in a constant voltage or constant current regime by the signals on lead 109. In the constant voltage mode the output from the waveform generator 136 is applied to the power amplifier 138. The constant current mode will be addressed later in this document. The power amplifier 138 is a unity gain amplifier capable of a peak output voltage of 10 Volts and a peak output current of 2.5 Amps. The current detector 139 senses the current delivered to the defect, line 99, and has an output voltage proportional to the current. This signal is precision full wave rectified by the full wave rectifier 145 and filtered by lowpass filter 147 to provide a voltage proportional to the average current through the defect. This average current is displayed on a digital voltmeter 149 as well as being fed back over lead 116 to the computer controller 121 and difference amplifier 143. In a similar manner, the voltage across the defect is passed through a full wave rectifier 146 and lowpass filter 148 to obtain a voltage equal to the average value of the defect voltage. This signal is displayed on digital voltmeter 150 and fed back to the computer controller 121.

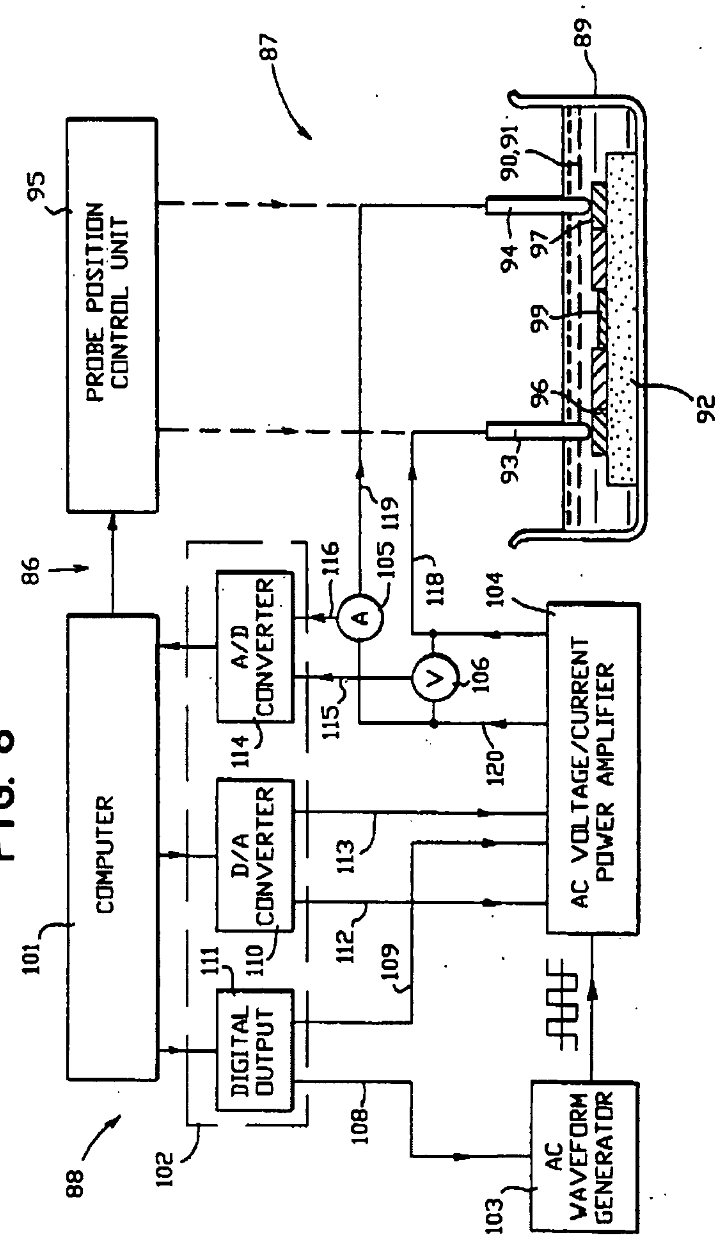

FIG. 8

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3	US 6004440 A	36	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
4	US 5695630 A	7	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
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7	US 5091152 A	11	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

US-PAT-NO: 6322674

DOCUMENT-IDENTIFIER: US 6322674 B1

TITLE: Cathode current control system for a wafer electroplating apparatus

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Detailed Description Text - DETX (11):
In the feedback system 185, a current monitor circuit 190 monitors the current flowing through the respective segment 130 and provides a signal indicative of the magnitude of the current to a central processing unit 195. The control processing unit 195, in turn, provides a feedback signal to a bias control circuit 200 that generates an output voltage therefrom to the inputs of comparator 180. Comparator 180 uses the signal from the bias control circuit 200 and, further, from a plating waveform generator 205 to generate the drive signal to the gate terminals of the FETs 170 and 175.

Current US Original Classification - CCOR (1):
204/212

Current US Cross Reference Classification - CCXR (1):
204/218

Current US Cross Reference Classification - CCXR (2):
204/242

(12) **United States Patent**
Berner et al.

(10) Patent No.: **US 6,322,674 B1**
(45) Date of Patent: **Nov. 27, 2001**

(54) **CATHODE CURRENT CONTROL SYSTEM FOR A WAFER ELECTROPLATING APPARATUS**

(75) Inventors: **Robert W. Berner**, Eagle, ID (US); **Joseph J. Fatula, Jr.**, San Jose, CA (US); **Robert Hitzfeld**, San Jose, CA (US); **Richard Contreras**, San Jose, CA (US); **Andrew Chiu**, Milpitas, CA (US)

(73) Assignee: **Semitool, Inc.**, Kalispell, MT (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/440,761**

(22) Filed: **Nov. 16, 1999**

Related U.S. Application Data

(62) Division of application No. 08/933,450, filed on Sep. 18, 1997, now Pat. No. 6,004,440.

(51) Int. Cl.⁷ **C25D 17/00**
(52) U.S. Cl. **204/212**; 204/218; 204/242
(58) Field of Search 204/212, 218, 204/242

(56) **References Cited**

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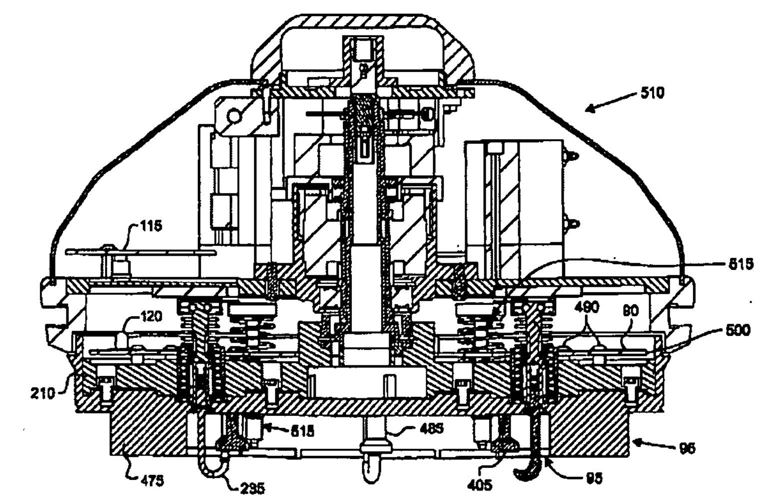
Primary Examiner—Donald R. Valentine
Assistant Examiner—Erica Smith-Hicks
(74) *Attorney, Agent, or Firm*—Polit & Associates, L.L.C.

(57) **ABSTRACT**

A cathode current control system employing a current thief for use in electroplating a wafer is set forth. The current thief comprises a plurality of conductive segments disposed to substantially surround a peripheral region of the wafer. A first plurality of resistance devices are used, each associated with a respective one of the plurality of conductive segments. The resistance devices are used to regulate current through the respective conductive finger during electroplating of the wafer. Various constructions are used for the current thief and further conductive elements, such as fingers, may also be employed in the system. As with the conductive segments, current through the fingers may also be individually controlled. In accordance with one embodiment of the overall system, selection of the resistance of each respective resistance device is automatically controlled in accordance with predetermined programming.

8 Claims, 28 Drawing Sheets



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EAST Browser - L6: (11) 5 not 3 | US 5091152 A | Tag: S | Doc: 7/11 (SORTED) | Format : KWIC

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3	US 6004440 A	36	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
4	US 5695630 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
5	US 5336379 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
6	US 5287060 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
7	US 5091152 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5091152

DOCUMENT-IDENTIFIER: US 5091152 A

TITLE: Apparatus for electrically destroying targeted organisms in fluids

---------- KWIC ---------

Detailed Description Text - DETX (11):
A waveform generator 6, and a current regulator 8 are provided. The outputs of the waveform generator 6 and the current regulator 8 are supplied to an amplifier 7.

Detailed Description Text - DETX (12):
The waveform generator 6 is capable of providing a variable frequency output in a sine, square, pulsed negative-pulsed positive triggered, or ~~saw tooth~~ waveform output 12, so that the targeted organism in inner area 33 ~~and~~ outer area 34, as shown in FIG. 2. can be destroyed.

Detailed Description Text - DETX (15):
As shown in FIG. 1B, a power supply 17 is provided which is capable of AC or DC operation. or in an uninterrupted configuration. The power output path to the waveform generator 6, the current regulator 8 and the power amplifier 7 are shown as 14.

Detailed Description Text - DETX (25):
FIGS. 6A and 6B show a second embodiment of the present invention. In this embodiment, the power supply 17. waveform generator 6, current regulator 8, amplifier 7 and transformer 5 are configured as shown in FIG. 1, except that the output of the transformer 5 is connected to the chamber in a single ended output arrangement with the center tap (at 10 and 11) not used. This embodiment is for static sterilization purposes using a two electrode chamber.

Claims Text - CLTX (9):
A.C. current waveform generator means for generating a plurality of waveforms with variable frequency and magnitude;

Claims Text - CLTX (11):
amplifier means, connected to receive the outputs of said waveform generator means and said current regulator means, for providing an amplified current;

Claims Text - CLTX (12):
power supply means for supplying power to said waveform generator means, said current regulator means, said amplifier means and said transformer means; and

Claims Text - CLTX (14):

FIG. 4

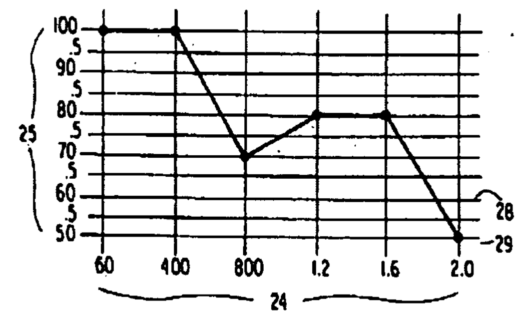

FIG. 5

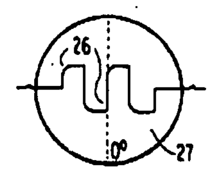

FIG. 6A

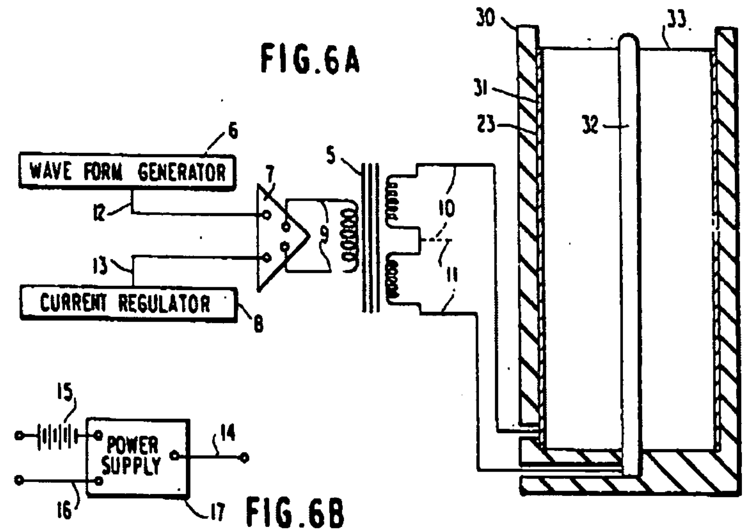

FIG. 6B

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  - L2: (4611) waveform adj generator
  - L3: (12) l1 and l2
  - L4: (16883) (204/198-297.16).CCLS.
  - L5: (15) l2 and l4
  - L6: (11) l5 not l3
  - L7: (187378) triangular or triangle
  - L8: (25336) sawtooth or (saw adj tooth)
  - L9: (207945) l7 or l8
  - L10: (451256) wave or waves or waveform or waveforms
  - L11: (16752) l9 near2 l10
  - L12: (59) l1 and l11
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EAST Browser - L12: (59) 1 and 11 | US 6143158 A | Tag: S | Doc: 10/59 (SORTED) | Format : KWIC

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	Document ID	Pages	1		3			U	S	C	P	Kind Codes	Sou
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6	US 6346182 B1	15							☑				USPAT
7	US 6334945 B1	11							☑				USPAT
8	US 6174425 B1	10							☑				USPAT
9	US 6171952 B1	9							☑				USPAT
10	US 6143158 A	27							☑				USPAT
11	US 6103087 A	29							☑				USPAT

US-PAT-NO: 6143158

DOCUMENT-IDENTIFIER: US 6143158 A

TITLE: Method for producing an aluminum support for a lithographic printing plate

---------- KWIC ---------

Detailed Description Text - DETX (135):

As to AC power supply waveforms used in electrochemical surface roughening, a sine wave, a rectangular wave, a trapezoidal wave and a triangular wave can be sued. A rectangular wave or a trapezoidal wave as shown in FIG. 9 is preferred, and a trapezoidal wave is particularly preferred.

Current US Original Classification - CCOR (1):
205/219

United States Patent [19]

Nishino et al.

[11] Patent Number: 6,143,158

[45] Date of Patent: Nov. 7, 2000

[54] METHOD FOR PRODUCING AN ALUMINUM SUPPORT FOR A LITHOGRAPHIC PRINTING PLATE

[75] Inventors: Atsuo Nishino; Yoshitaka Masuda; Akio Uesugi, all of Shizuoka, Japan

[73] Assignee: Fuji Photo Film Co., Ltd., Minami-Ashigara, Japan

[21] Appl. No.: 09/063,727

[22] Filed: Apr. 24, 1998

[30] Foreign Application Priority Data

Apr. 25, 1997 [JP] Japan 9-109528
Jun. 23, 1997 [JP] Japan 9-166143

[51] Int. Cl.[7] C25D 5/34; B23H 11/00; C25F 3/00

[52] U.S. Cl. 205/219; 205/658; 205/660; 205/662; 205/672; 205/674

[58] Field of Search 205/658, 659, 205/660, 661, 672, 674, 219, 220, 214, 212, 662

[56] References Cited

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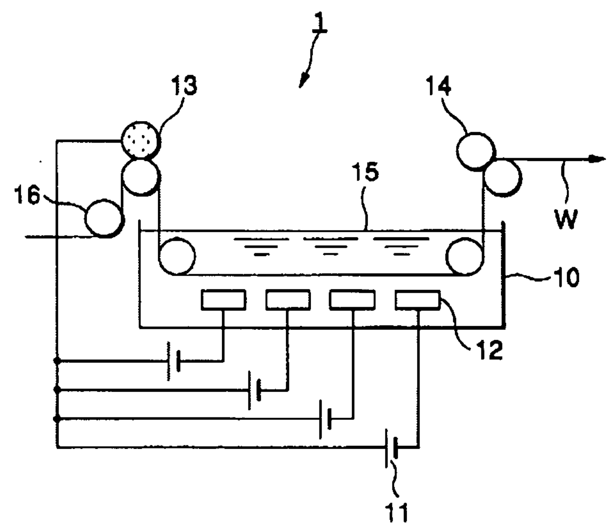
9-277735	10/1997	Japan .

Primary Examiner—Donald R. Valentine
Attorney, Agent, or Firm—Burns, Doane, Swecker & Mathis, LLP

[57] ABSTRACT

A method for producing an aluminum support for a lithographic printing plate comprising the steps of (a) electrolytic polishing an aluminum plate in an alkaline aqueous solution; and (b) electrochemically surface roughening the aluminum plate using direct or alternating current in an acidic aqueous solution in this order, and also a method for producing an aluminum support for a lithographic printing plate comprising an electrolytic polishing step of treating an aluminum plate used as an anode in an alkaline aqueous solution at a current density of 5 A/dm^2 to 200 A/dm^2 while allowing the alkaline aqueous solution to flow between the aluminum plate and an electrode at an average flow rate of 10 cm/second to 400 cm/second.

24 Claims, 8 Drawing Sheets



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13	US 5935408 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
14	US 5865978 A	21	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
15	US 5837121 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
16	US 5832834 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
17	US 5804400 A	16	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
18	US 5788819 A	48	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
19	US 5705230 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5705230

DOCUMENT-IDENTIFIER: US 5705230 A

TITLE: Method for filling small holes or covering small recesses in the surface of substrates

---------- KWIC ---------

Detailed Description Text - DETX (3):

In the step of deposition, a controlled varying voltage and/or an energy such as low-frequency, high-frequency or ultrasonic vibrations or a light beam is preferably applied to the substrate. The applicable varying voltage includes voltages of pulsed waveforms such as square wave, trapezoidal wave, triangular wave, sinusoidal wave and random wave. The voltages of such pulsed waveforms may have both positive and negative polarities or they may have only negative polarity. The repetitive voltage components may be zero at periodic times; alternatively, a d.c. component may be added to a periodic wave. The value of the voltage to be applied is preferably such that a maximum absolute value of current density on the surface of the substrate Is in the range of from 10.sup.-3 to 1 A/cm.sup.2. The low-frequency, high-frequency or ultrasonic vibrations are preferably such that their frequency is in the range of from 45 Hz to 2.5 GHz.

Current US Cross Reference Classification - CCXR (1):
205/104

Current US Cross Reference Classification - CCXR (2):
205/191

Current US Cross Reference Classification - CCXR (3):
205/91

US005705230A

United States Patent [19]

Matanabe et al.

[11] Patent Number: 5,705,230

[45] Date of Patent: Jan. 6, 1998

[54] METHOD FOR FILLING SMALL HOLES OR COVERING SMALL RECESSES IN THE SURFACE OF SUBSTRATES

[75] Inventors: Toru Matanabe, Hopewell Junction, N.Y.; Hirokazu Ezawa, Tokyo, Japan; Masahiro Miyata, Chiba-ken, Japan; Yukio Ikeda, Tokyo, Japan; Manabu Tsujimura, Kanagawa-ken, Japan; Hiroaki Inoue, Kanagawa-ken, Japan; Takeyuki Odaira, Kanagawa-ken, Japan; Naoaki Ogure, Tokyo, Japan

[73] Assignee: Ebara Corporation, Tokyo, Japan

[21] Appl. No.: 324,460

[22] Filed: Oct. 17, 1994

Related U.S. Application Data

[63] Continuation of Ser. No. 32,657, Mar. 17, 1993, abandoned.

[30] Foreign Application Priority Data

Mar. 17, 1992 [JP] Japan 4-091778

[51] Int. Cl.[6] B05D 1/18

[52] U.S. Cl. 427/438; 205/91; 205/104; 205/191

[58] Field of Search 205/91, 118, 161, 205/191; 427/437, 438, 443.1, 595

[56] References Cited

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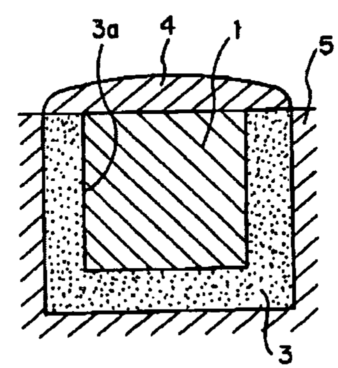
Primary Examiner—John Niebling
Assistant Examiner—Brendan Mee
Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt, P.C.

[57] ABSTRACT

The improved method comprises contacting a substrate 5 at least once by a liquid containing the elements that compose a pure metal or an alloy with which the small holes or recesses 3a in the substrate 5 are to be filled or covered, whereby the liquid wets the inner surfaces of said small holes or recesses 3a while, at the same time, said pure metal or said alloy is deposited on the surface of said substrate 5. The method is capable of filling small holes or covering small recesses in the surface of the substrate 5 with improved efficiency while, at the same time, it improves the heat resistance and materials stability of the part that contains the formed filling or covering layer.

3 Claims, 4 Drawing Sheets



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EAST Browser - L12: (59) 1 and 11 | US 5437779 A | Tag: S | Doc: 22/59 (SORTED) | Format : KWIC

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17	US 5804400 A	16	☐	☑	☐	☐		USPAT
18	US 5788819 A	48	☐	☑	☐	☐		USPAT
19	US 5705230 A	9	☐	☑	☐	☐		USPAT
20	US 5662788 A	6	☐	☑	☐	☐		USPAT
21	US 5616229 A	8	☐	☑	☐	☐		USPAT
22	US 5437779 A	7	☐	☑	☐	☐		USPAT

US-PAT-NO: 5437779

DOCUMENT-IDENTIFIER: US 5437779 A

TITLE: Method of making a magnetic record medium

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Drawing Description Text - DRTX (2):

FIGS. 1(A) through 1(D) show examples of alternating waveform currents employed in the present invention, wherein FIG. 1(A) designates a sinusoidal waveform, FIG. 1(B), a rectangular waveform, FIG. 1(C), a trapezoidal waveform, and FIG. 1(D), a triangular waveform.

Detailed Description Text - DETX (4):

In this invention, the textured surface of the substrate is electrolyzed in an electrolyte of an acidic solution by applying an electric potential on the substrate. As an electrolyte, for instance, an aqueous solution of at least one selected from the group consisting of sulfuric acid, nitric acid, hydrochloric acid, chromic acid, phosphoric acid, oxalic acid, and acetic acid in a range of concentration of 0.5 through 40 weight %, preferably 1 through 30 weight %, and particularly phosphoric acid is preferable. As for the electrolytic conditions, it is preferable that the substrate is treated wherein an electric potential is applied on the substrate in the electrolyte, the solution temperature is in a range of 10.degree. through 70.degree. C., the current density is in a range of 0.1 through 50 mA/cm.sup.2, preferably 0.5 through 45 mA/cm.sup.2, more preferably 1.0 through 20 mA/cm.sup.2, the electrolytic time is in a range of 1 through 400 seconds, preferably 2 through 200 seconds, and the quantity of electricity (a product of current density by electrolytic time) is in a range of 10 through 1000 mA.multidot.second/cm.sup.2, preferably 50 through 600 mA.multidot.second/cm.sup.2. The electric potential applied in the electrolysis reaction is a direct current or an alternating waveform current wherein the polarities are alternately reversed, and especially the alternating waveform current is preferable. When a direct current potential is employed, it is preferable to perform the treatment with current density in a range of 1.0 through 25 mA/cm.sup.2. The alternating waveform current is provided by alternately reversing (converting) the positive and negative polarities, that is, the polarities of anode and cathode, which is, for instance, a single phase alternating current of a sinusoidal wave, a three-phase alternating current of a sinusoidal wave, a rectangular wave, a triangular wave, a trapezoidal wave or the like. The frequency of the alternating waveform current is not smaller than 0.1 and smaller than 500 Hz, preferably in a range of 0.1 through 300 Hz, more preferably in a range of 0.5 through 200 Hz. Further, it is preferable to select a ratio (Q.sub.a /Q.sub.c) of the quantity of electricity in anode time (Q.sub.a) over the quantity of electricity in cathode time (Q.sub.c) in case of employing the alternating waveform current, is in a range of 0 through 2.0, preferably 0.8 through 1.5, more preferably 0.9 through 1.1.

Current US Original Classification - CCOR (1):
205/106

US005437779A

United States Patent [19]

Shige et al.

[11] Patent Number: 5,437,779

[45] Date of Patent: Aug. 1, 1995

[54] METHOD OF MAKING A MAGNETIC RECORD MEDIUM

[75] Inventors: Tomoo Shige; Yasushi Makabe; Masataka Yokoyama, all of Kurashiki, Japan

[73] Assignee: Mitsubishi Chemical Corporation, Tokyo, Japan

[21] Appl. No.: 163,551

[22] Filed: Dec. 9, 1993

[30] Foreign Application Priority Data

Dec. 11, 1992 [JP] Japan 4-331665

[51] Int. Cl.[6] C25D 5/34

[52] U.S. Cl. 205/106; 204/192.2; 205/201; 205/206; 205/214; 205/219; 427/129

[58] Field of Search 205/206, 214, 219, 198, 205/199, 200, 201, 106, 107; 204/192.2; 427/129

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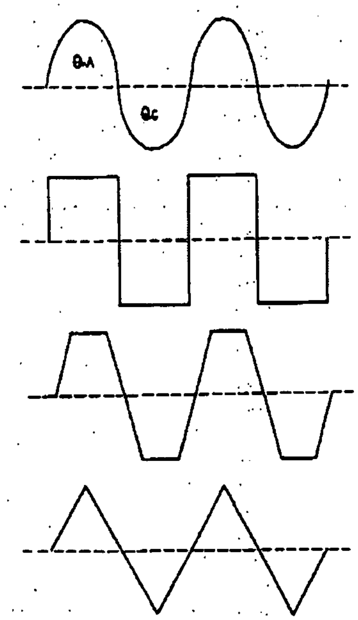
Primary Examiner—John Niebling
Assistant Examiner—Edna Wong
Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt

[57] ABSTRACT

A method of making a magnetic record medium comprising the steps of: texturing a surface of a substrate by a first surface treatment; electrolyzing the surface of the textured substrate in an electrolyte of an acidic solution by applying an electric potential on the nonmagnetic substrate by a second surface treatment; and forming an undercoating layer and a magnetic layer on the surface of the electrolyzed substrate.

17 Claims, 1 Drawing Sheet



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19	US 5705230 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
20	US 5662788 A	6	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
21	US 5616229 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
22	US 5437779 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
23	US 5384215 A	15	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
24	US 5364522 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5364522

DOCUMENT-IDENTIFIER: US 5364522 A

TITLE: Boride, carbide, nitride, oxynitride, and silicide infiltrated electrochemical ceramic films and coatings and the method of forming such

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Detailed Description Text - DETX (8):
A substrate subject to anodic coating is direct current positively polarized. A substrate subject to cathodic coating is direct current negatively polarized. An external voltage is connected across the two electrodes, a DC, or a pulsed DC, or a sine wave DC, or a sawtooth DC, or a triangular DC, or a stepped DC, and or a mixture thereof is passed through the electrodes and electrolytes.

Detailed Description Text - DETX (9):
This invention relates that the preferred electric potentials or current waveforms consists of a pure DC, a pulsed DC, a sine wave DC, a sawtooth DC, a triangular DC, a stepped DC, a sine wave AC, and a mixture thereof.

Detailed Description Text - DETX (11):
When a pulsed DC voltage, or a sine wave DC, or a sawtooth DC, or a triangular DC, or a stepped DC is applied, different ions will be coated at different potentials to form a layered ceramic precursor coating. A ceramic precursor composition with layered, laminal ultrastructures including superlattices are obtained by the modulating of the applied voltage or current. A multilayered, or laminal ultrastructures including a superlattice precursor coating can be obtained by the modulating of the applied voltage or current densities at the proper waveform or pulse frequency. An amplitude-modulated DC voltage or current density provides a finely controlled layered ceramic precursor composition. The invention also relates to the discovery that following an infiltration the multilayered and superlattice ceramic precursor coatings are infiltrated to multilayered and superlattice ceramic coatings having boride, carbide, nitride, oxynitride and silicide infiltrated concentration gradients with the maximum at surface and minimum in bulk of the parent ceramic coatings.

Claims Text - CLTX (10):
10. A method as claimed in claim 7 wherein said amplitude-modulated electric current is an electric current with a constant or time-variated waveform selected from the group consisting of a single pulse, a multiple pulse, a sine wave, a sawtooth, a triangular, a stepped shape, a convolution of different geometric shapes, and a mixture thereof.

Claims Text - CLTX (20):
20. A method as claimed in claim 16 wherein said amplitude-modulated electric current is an electric current with a constant or time-variated waveform selected from the group consisting of a single pulse, a multiple

US005364522A

United States Patent [19]

Wang

[11] Patent Number: 5,364,522

[45] Date of Patent: Nov. 15, 1994

[54] BORIDE, CARBIDE, NITRIDE, OXYNITRIDE, AND SILICIDE INFILTRATED ELECTROCHEMICAL CERAMIC FILMS AND COATINGS AND THE METHOD OF FORMING SUCH

[76] Inventor: Liang Wang, 5129 Maddox Rd., Tallahassee, Fla. 32303

[21] Appl. No.: 35,424

[22] Filed: Mar. 22, 1993

[51] Int. Cl.[5] C25D 5/50; C25D 3/02

[52] U.S. Cl. 205/50; 205/108; 205/224; 205/118; 205/229; 208/162; 208/174; 208/316; 428/446; 428/689; 428/688; 428/698; 428/704

[58] Field of Search 205/59, 106, 108, 224, 205/229, 118, 174, 316, 162; 428/446, 689, 688, 698, 702

[56] References Cited

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J. Electrochem. Soc., 1992, 139, 67–69.
J. Electrochem. Soc., 1980, 127, 2053–2057.
Chem. Abstracts; vol. 57-Ceramics; p. 265; Abst. 119066g.

Primary Examiner—Kathryn Gorgos

[57] ABSTRACT

Ceramic films and coatings, single or multi-layered, including superlattice, infiltrated with boride, carbide, nitride, oxynitride, and silicide were formed by methods which comprises of an electrochemical coating of a ceramic precursor by a constant or an amplitude-modulated electric current with a DC component in a medium containing at least one of the ionic species for the composition of the ceramic precursor, following single or multiple infiltration in a medium containing at least one of the compounds selected from a B-containing compounds, a C-containing compounds, a N-containing compounds, a Si-containing compounds, and a mixture thereof, by heating means selected from radio-frequency, microwave, thermal, flame, plasma, laser, and a mixture thereof.

24 Claims, No Drawings

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23	US 5384215 A	15	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
24	US 5364522 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
25	US 5320719 A	10	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
26	US 5268235 A	10	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
27	US 5171416 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
28	US 5158653 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
29	US 5147515 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5147515

DOCUMENT-IDENTIFIER: US 5147515 A

TITLE: Method for forming ceramic films by anode-spark discharge

---------- KWIC ---------

Brief Summary Text - BSTX (31):
The output from a power supply may be a direct current having any wave form, but preferably those having pulse shape (rectangular wave form), saw-tooth wave form or DC half-wave form.

Current US Cross Reference Classification - CCXR (1):
205/320

Current US Cross Reference Classification - CCXR (2):
205/321

Current US Cross Reference Classification - CCXR (3):
205/322

Current US Cross Reference Classification - CCXR (4):
205/323

US005147515A

United States Patent [19]

Hanagata et al.

[11] Patent Number: **5,147,515**

[45] Date of Patent: **Sep. 15, 1992**

[54] **METHOD FOR FORMING CERAMIC FILMS BY ANODE-SPARK DISCHARGE**

[75] Inventors: Haruo Hanagata, Ebina; Tsukasa Suzuki; Kazuo Yanagida, both of Kasukabe; Hidesato Igarashi, Tokyo, all of Japan

[73] Assignee: Dipsol Chemicals Co., Ltd., Tokyo, Japan

[21] Appl. No.: 573,703

[22] Filed: Aug. 28, 1990

[30] Foreign Application Priority Data

Sep. 4, 1989 [JP] Japan 1-228639
Mar. 6, 1990 [JP] Japan 2-54827

[51] Int. Cl.[5] H05F 3/04

[52] U.S. Cl. 204/164; 205/320; 205/321; 205/322; 205/323; 427/37

[58] Field of Search 204/56.1, 58, 58.4, 204/164; 427/37; 106/628, 635, 637; 264/22; 205/321, 322, 323, 320

[56] References Cited

U.S. PATENT DOCUMENTS

3,812,021 5/1974 Craig et al. 204/58
3,812,022 5/1974 Rogers et al. 204/58
3,832,293 8/1974 Hradcovsky et al. 204/58.4
3,956,080 5/1976 Hradcovsky et al. 204/58.4
3,960,676 6/1976 Miyosawa et al. 205/323

FOREIGN PATENT DOCUMENTS

151330 10/1981 Fed. Rep. of Germany .
156003 7/1982 Fed. Rep. of Germany .

Primary Examiner—John Niebling
Assistant Examiner—Brian M. Bolam
Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt

[57] **ABSTRACT**

A method for forming a ceramics film on the surface of a substrate comprises performing spark discharge in an electrolytic bath, wherein the electrolytic bath comprises an aqueous solution of a water-soluble or colloidal silicate and/or an oxyacid salt to which ceramics fine particles and/or specific fine particles are dispersed and the spark discharge is carried out in the electrolytic bath while ensuring the suspended state of the ceramics particles and/or the specific fine particles in the electrolytic bath. The method makes it possible to effectively form, on the surface of a metal substrate, ceramics films having a variety of color tones as well as excellent insulating properties and hardness. Moreover, it further makes it possible to effectively form a composite ceramics film having excellent wear resistance on the surface of a metal substrate.

13 Claims, No Drawings

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29	US 5147515 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5147515

DOCUMENT-IDENTIFIER: US 5147515 A

TITLE: Method for forming ceramic films by anode-spark discharge

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Current US Cross Reference Classification - CCXR (3):
205/322

Current US Cross Reference Classification - CCXR (4):
205/323

US005147515A

United States Patent [19]

Hanagata et al.

[11] Patent Number: 5,147,515

[45] Date of Patent: Sep. 15, 1992

[54] METHOD FOR FORMING CERAMIC FILMS BY ANODE-SPARK DISCHARGE

[75] Inventors: Haruo Hanagata, Ebina; Tsukasa Suzuki; Kazuo Yanagida, both of Kasukabe; Hidesato Igarashi, Tokyo, all of Japan

[73] Assignee: Dipsol Chemicals Co., Ltd., Tokyo, Japan

[21] Appl. No.: 573,703

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[52] U.S. Cl. 204/164; 205/320; 205/321; 205/322; 205/323; 427/37

[58] Field of Search 204/56.1, 58, 58.4, 204/164; 427/37; 106/628, 635, 637; 264/22; 205/321, 322, 323, 320

[56] References Cited

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3,956,080 5/1976 Hradcovsky et al. 204/58.4
3,960,676 6/1976 Miyosawa et al. 205/323

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151330 10/1981 Fed. Rep. of Germany .
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Primary Examiner—John Niebling
Assistant Examiner—Brian M. Bolam
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[57] ABSTRACT

A method for forming a ceramics film on the surface of a substrate comprises performing spark discharge in an electrolytic bath, wherein the electrolytic bath comprises an aqueous solution of a water-soluble or colloidal silicate and/or an oxyacid salt to which ceramics fine particles and/or specific fine particles are dispersed and the spark discharge is carried out in the electrolytic bath while ensuring the suspended state of the ceramics particles and/or the specific fine particles in the electrolytic bath. The method makes it possible to effectively form, on the surface of a metal substrate, ceramics films having a variety of color tones as well as excellent insulating properties and hardness. Moreover, it further makes it possible to effectively form a composite ceramics film having excellent wear resistance on the surface of a metal substrate.

13 Claims, No Drawings

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US-PAT-NO: 4923574

DOCUMENT-IDENTIFIER: US 4923574 A

TITLE: Method for making a record member with a metallic antifriction overcoat

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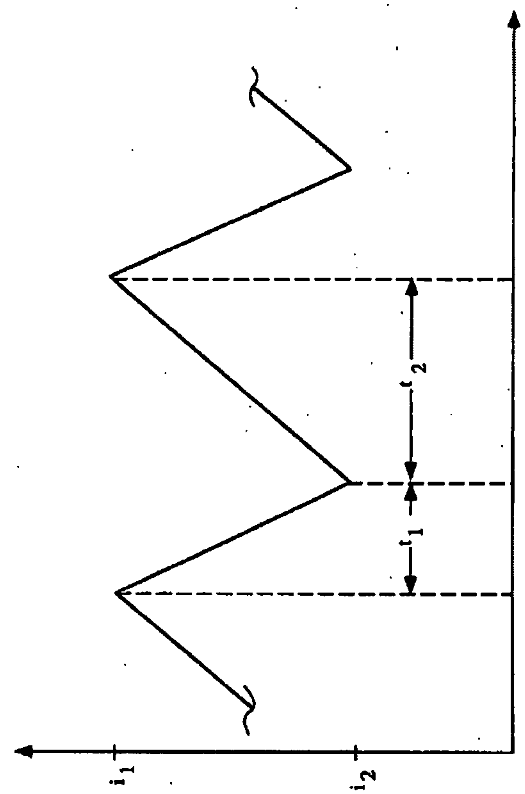
Brief Summary Text - BSTX (19):
If desired, the plating current density (or cathode potential) used to produce the multilayered structure may be selected to have a stepped, triangular, or sinusoidal waveform which is a periodic function of time. Once the plating bath is selected the composition and thickness of each layer is selected independently by controlling the current density (or cathode potential), as a function of time.

Drawing Description Text - DRTX (8):
FIG. 7 shows a triangular or swept waveform for applied current density as a function of time.

Detailed Description Text - DETX (43):
It should be noted that the duration of the high or low values of current density as illustrated by the waveform shown in FIG. 5 can be varied and controlled by the function generator to achieve desired thickness of the layers L1, ... LN. The thickness of each layer is directly related to the amount of electric charge utilized during its electrodeposition. Thus, for a given current density, the thickness of a layer is controlled by the time the current is applied to the electrolytic cell. The composition of the plated alloy can be varied and controlled by changing the current density (or cathode potential) to a value which gives the desired composition. FIG. 7 provides another example of a graph of a current density versus time which may be used to generate another layered structure. A similar waveform may be applied to the cathode potential. The result of the use of this triangular or swept waveform is a triangular or graded variation in the composition of the alloy layers plated.

Detailed Description Text - DETX (44):
FIGS. 5 and 7 are merely examples of the many waveforms which may be employed in order to form a layered structure. Other alternative embodiments of the present invention include electrodepositing more than two layers per cycle. This is accomplished by creating a series of different current densities or cathode potentials before repeating the cycle. Thus, it can be seen that the method of this invention permits the design of a resulting plated structure by variation of the magnitude of the current or cathode potential applied and the duration that a given current density or cathode potential is applied. In particular, the waveform shown in FIG. 8 may be used to produce four layers per cycle: a first layer of graded composition during time period t.sub.1 ; a second layer of constant composition during time period t.sub.2 ; a third layer of graded composition during time period t.sub.3 ; and a fourth layer of constant composition during time period t.sub.4. Similarly, other combinations of stepped, triangular, and sine waveforms may also be utilized.

U.S. Patent May 8, 1990 Sheet 7 of 8 4,923,574



CURRENT DENSITY (mA/cm²)

TIME (SECONDS)

FIG. 7

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42	US 4704196 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
43	US 4656411 A	18	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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45	US 4490218 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
46	US 4468293 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 4468293

DOCUMENT-IDENTIFIER: US 4468293 A

TITLE: Electrochemical treatment of copper for improving its bond strength

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Brief Summary Text - BSTX (20):

More particularly, an electrolytic bath comprising a copper sulfate-sulfuric acid solution is maintained in an electrochemical cell. The cell has an anode and a cathode. The cathode comprises the copper sheet or foil upon which the dendrites are to be deposited. Current is applied across the cell either by a constant current source and a function generator or a constant voltage source and a function generator. The applied current preferably has a suitable wave form, such as a square wave, a triangular wave, a sinusoidal wave, etc. The applied current causes clusters of copper particles to be deposited on and bonded to the copper sheet or foil. These clusters of copper particles from the dendrites. They generally have a relatively fine structure which is highly desirable. It is believed that the relatively fine dendritic structure is a result of the initiation of many nucleation sites during an initial current pulse and the renucleation of the dendritic structures each time there is another current pulse. In addition, undesirable columnar structures are avoided by not exposing the dendritic structures to relatively long periods of time at current densities above the limiting current density. After the sheet or foil has been treated in accordance with the instant invention, it may be laminated to a non-metallic substrate so as to form, for example, a printed circuit laminate.

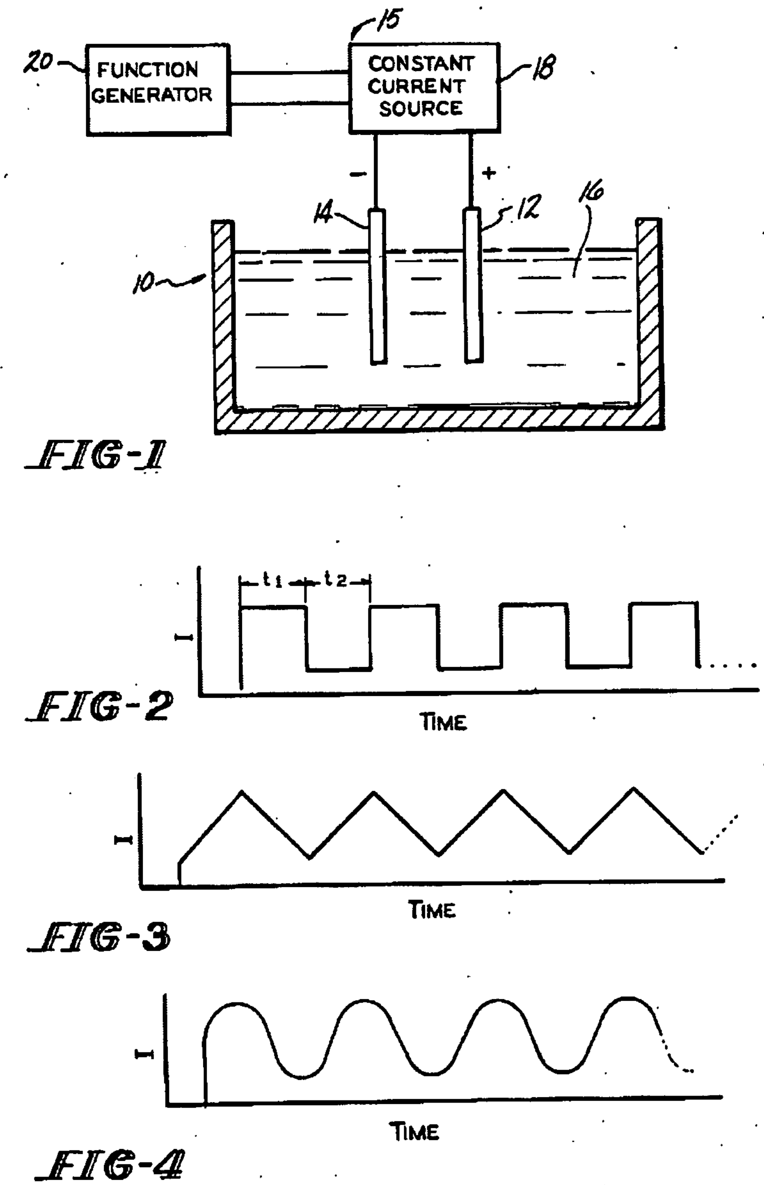
Detailed Description Text - DETX (8):

The current applying system 15 preferably comprises a constant d-c current source 18 and a function generator 20. The function generator 20 provides the current applied to the cell 10 with a desired waveform. The current applied to the cell 10 is preferably an uninterrupted, multi-cycle, fluctuating current having a cathodic portion with first and second current densities each with a magnitude greater than zero and flowing in only one direction. As shown in FIGS. 2-4, the applied current is a non-zero base cathodic current in which the second current density is also the base current density. Any suitable current waveform, such as the square waveform shown on FIG. 2, the triangular waveform shown in FIG. 3, and the sinusoidal waveform shown in FIG. 4 may be used as long as it has a cathodic portion with first and second current densities having a magnitude greater than zero. The constant current source 18 and the function generator 20 may comprise, respectively, any conventional constant current source and function generator as are known in the art.

Detailed Description Text - DETX (17):

The voltage applied to the cell 10' may have any suitable waveform such as a square waveform, a triangular waveform, a sinusoidal waveform, etc. Constant voltage source 28 and function generator 30 may comprise any conventional constant voltage source and function generator as are known in the art. The voltage control system 25 will obtain substantially the same result as the current applying system 15.

U.S. Patent Aug. 28, 1984 Sheet 1 of 3 4,468,293



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955	US 4338187 A	12	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
956	US 4338176 A	5	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 4338176

DOCUMENT-IDENTIFIER: US 4338176 A

TITLE: System for generating and autocontrolling the voltage or current wave form applicable to processes for the electrolytic coloring of anodized aluminium

---------- KWIC ---------

Assistant Examiner - XA (1):
Leader; William

United States Patent [19]

García Peláez

[11] **4,338,176**

[45] **Jul. 6, 1982**

[54] SYSTEM FOR GENERATING AND AUTOCONTROLLING THE VOLTAGE OR CURRENT WAVE FORM APPLICABLE TO PROCESSES FOR THE ELECTROLYTIC COLORING OF ANODIZED ALUMINIUM

[75] Inventor: Jose' Garcia Pelaez, Alicante, Spain

[73] Assignee: Empresa Nacional del Aluminio, S.A. (ENDASA), Madrid, Spain

[21] Appl. No.: 83,943

[22] Filed: Oct. 11, 1979

[30] Foreign Application Priority Data

Oct. 31, 1978 [ES] Spain 474,736

[51] Int. Cl.[3] C25D 11/22; G05F 1/44

[52] U.S. Cl. 204/228; 204/DIG. 9; 323/281; 330/265; 363/97

[58] Field of Search 204/228-231, 204/DIG. 9, 204, 205, 211, 218, 223; 363/97; 330/259, 265, 271, 293, 297; 323/273, 281

[56] References Cited

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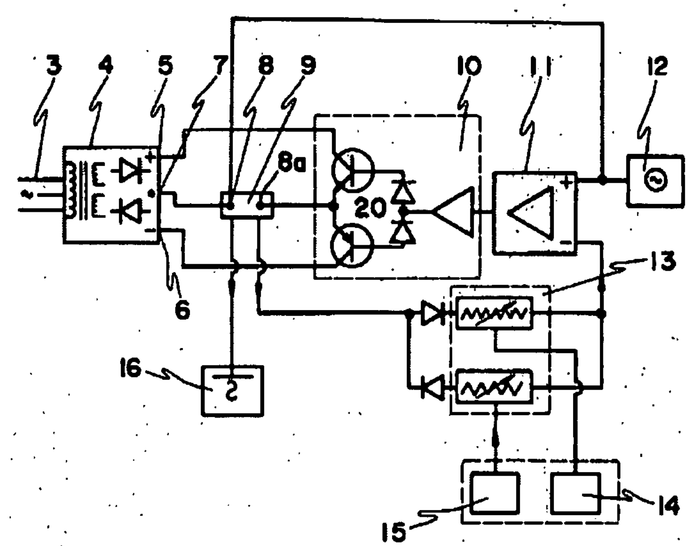
Primary Examiner—Delbert E. Gantz
Assistant Examiner—William Leader
Attorney, Agent, or Firm—Wenderoth, Lind & Ponack

[57] ABSTRACT

A system for generating and autocontrolling the voltage or current wave form in a process for the electrolytic coloring of anodized aluminium includes feeding power from a source of symmetrical direct current, with a neutral connection directly coupled to a load, while the positive and negative voltages supplied by the source pass through a power control stage which is controlled by a bipolar operational amplifier. The bipolar operational amplifier has two signal inputs, a positive or non-inverting input which is connected to a signal generator, and a negative or inverting input having a signal which corresponds to the signal which actually exists on the electrodes of the electrolytic bath. This signal is processed in a half-wave outer controller which is controlled by a programming system.

7 Claims, 3 Drawing Figures



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6	US 5364522 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
7	US 5147515 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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9	US 4879018 A	5	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 4879018

DOCUMENT-IDENTIFIER: US 4879018 A

TITLE: Low voltage anodizing process and apparatus

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Detailed Description Text - DETX (12):
Aluminum oxide was being anodically formed on an aluminum test panel (alloy 6061) as the load and while using 22% sulphuric acid as the electrolyte and lead cathodes and while using the previously mentioned 0/50/100/150 volt output DC power supply PS1, full anodizing current was suddenly attained while the anodizing process was at low (about 15 volts) voltages. The oscilloscope picture immediately changed from a complex (irregular, zig-zag, non-saw-tooth) to a clean wave form (that is, a rise/fall/off-time saw-tooth pattern comparable to that shown in FIG. 4) and remained clean throughout. We completed the full anodizing run by step-wise increases in voltage up to about 30 volts and achieved, with ease, 2 mils of coating (0.002 inches) of excellent quality in 50 minutes with no indication at all of sudden current increase as happens in the prior art.

Current US Original Classification - CCOR (1):
205/108

Current US Cross Reference Classification - CCXR (2):
205/115

Current US Cross Reference Classification - CCXR (3):
205/316

Current US Cross Reference Classification - CCXR (4):
205/325

Current US Cross Reference Classification - CCXR (5):
205/328

U.S. Patent Nov. 7, 1989 **4,879,018**

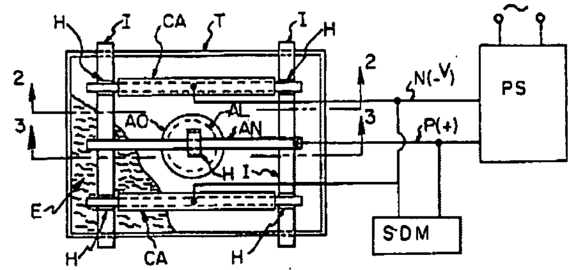

FIG.1

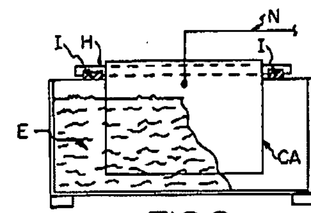

FIG.2

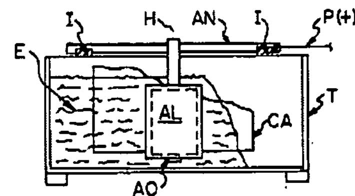

FIG.3

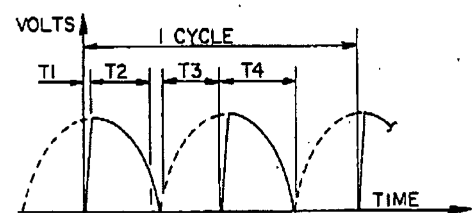

FIG.4

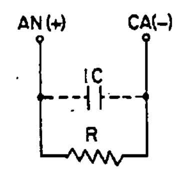

FIG 5

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File View Edit Tools Window Help

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  - L5: (30528) (wave adj form) or (wave adj forms)
  - L6: (178481) 14 or 15
  - L7: (6944) 13 same 16
  - L8: (16) 12 and 17
  - L9: (28957) ("205").CLAS.
  - L10: (45731) ("204").CLAS.
  - L11: (64358) 19 or 110
  - L12: (81) 111 and 17
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EAST Browser - L13: (65) 12 not 8 | US 6451181 B1 | Tag: S | Doc: 7/65 (SORTED) | Format : KWIC

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2	US 6554979 B2	18	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
3	US 6537433 B1	18	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
4	US 6506439 B1	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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6	US 6475351 B2	21	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
7	US 6451181 B1	15	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 6451181

DOCUMENT-IDENTIFIER: US 6451181 B1

TITLE: Method of forming a semiconductor device barrier layer

---------- KWIC ---------

Detailed Description Text - DETX (56):

FIG. 14 illustrates yet a third possible waveform 604 which may be used to form the barrier layer 220, of FIG. 10. Waveform 604 shows that pulsed power (either periodic or nonperiodic) is applied to the coil 52. When using a pulsed coil power waveform as in FIG. 14, alternating layers or portions of less tensile and more tensile tantalum can be incrementally deposited on the wafer 22 to form the barrier layer 220, of FIG. 10. Therefore, it has been determined that selectively powering on and off the coil at least once during the deposition process can be used to tune the stress of the barrier layer to accommodate a plurality of differing constraints or conditions. In addition, although FIG. 14 shows primarily step coverage wave forms, the waveforms that can be used on the coil, target, and/or wafer need not be step function curves over time. For example, it is possible to use triangle-shaped (sawtooth) waveforms, sinusoidal waveforms, logarithmic power curves, exponential power curves, combination thereof, or any other types of analog, continuous, or quantized wave forms to produce different types of stress characteristics of the tantalum (or refractory metal based) barrier layer 220 of FIG. 10. Alternatively, this processing methodology can be used with a variety of other conductive films, such metals, refractive metals, and refractive metal nitrides which may be prone to stress related problems. In addition, while less tensile and more tensile are used to described the relative stress of materials throughout this specification, those skilled in the art will appreciate that the terms less tensile and more compressive can be used interchangeably.

Current US Class - CLAS (1):
204

equate triangle = sawtooth

(12) **United States Patent**
Denning et al.

(10) Patent No.: **US 6,451,181 B1**
(45) Date of Patent: **Sep. 17, 2002**

(54) **METHOD OF FORMING A SEMICONDUCTOR DEVICE BARRIER LAYER**

(75) Inventors: Dean J. Denning, Del Valle; Sam S. Garcia, Austin; Bradley P. Smith, Austin; Daniel J. Loop, Austin; Gregory Norman Hamilton, Pflugerville; Md. Rabiul Islam; Brian G. Anthony, both of Austin, all of TX (US)

(73) Assignee: Motorola, Inc., Schaumburg, IL (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/261,879

(22) Filed: Mar. 2, 1999

(51) Int. Cl.[7] C23C 14/34

(52) U.S. Cl. 204/192.17; 204/192.15; 204/192.12; 204/192.3; 204/298.06; 438/582; 438/652; 438/653; 438/656; 438/685; 438/698

(58) Field of Search 204/192.12, 192.15, 204/192.17, 192.3, 298.08, 298.06; 438/582, 698, 652, 653, 656, 685

(56) **References Cited**

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WO	WO98/54377	12/1998

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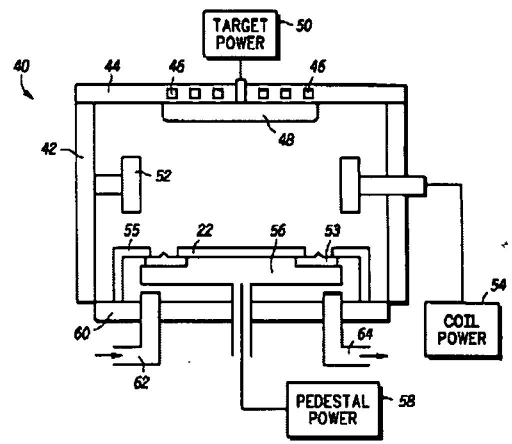
* cited by examiner

Primary Examiner—Rodney G. McDonald
(74) *Attorney, Agent, or Firm*—Robert A. Rodriguez; Keith E. Witek

(57) **ABSTRACT**

A method for forming an improved copper inlaid interconnect (FIG. 11) begins by performing an RF preclean operation (408) on the inlaid structure in a chamber (10). The RF preclean rounds corners (210a and 206a) of the structure to reduce voiding and improve step coverage while not significantly removing copper atoms from the underlying exposed copper interconnects surfaces (202a). A tantalum barrier (220) is then deposited where one portion of the tantalum barrier is more tensile than another portion of the tantalum barrier. After formation of the barrier layer (220), a copper seed layer (222) is formed over a top of the barrier layer. The copper layer is formed while clamping the wafer with an improved clamp (85) which reduces copper peeling and contamination at wafer edges. Copper electroplating and chemical mechanical polishing (CMP) processes are then used to complete the copper interconnect structure.

6 Claims, 6 Drawing Sheets



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28	US 5173167 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
29	US 5130002 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
30	US 5108846 A	10	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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32	US 5091152 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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34	US 5071527 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5071527

DOCUMENT-IDENTIFIER: US 5071527 A

TITLE: Complete oil analysis technique

---------- KWIC ---------

Detailed Description Text - DETX (3):

In performing a voltammetric analysis, the potential across the electrodes is varied linearly with time, and the resulting current is recorded as a function of the potential. A variation of this technique, known as cyclic voltammetric analysis, uses a potential variation as shown in FIG. 1. Initially, potential applied to the electrodes is of a first value E1 and is linearly increased over time to the second value E2. The potential is next reduced at the same rate until the potential again returns to E1. The potential continues to be reduced until it reaches a third value E3. The potential is then increased until it returns to E1, producing a sawtooth waveform. The cycle may then be repeated.

Current US Class - CLAS (1):
204

Current US Class - CLAS (2):
205

United States Patent [19]

Kauffman

[11] Patent Number: 5,071,527

[45] Date of Patent: Dec. 10, 1991

[54] COMPLETE OIL ANALYSIS TECHNIQUE

[75] Inventor: Robert E. Kauffman, Kettering, Ohio

[73] Assignee: University of Dayton, Dayton, Ohio

[21] Appl. No.: 545,842

[22] Filed: Jun. 29, 1990

[51] Int. Cl.⁵ G01N 27/26

[52] U.S. Cl. 204/153.1; 204/412; 204/434; 324/439; 436/60

[58] Field of Search 204/153.1, 412, 434; 324/439, 450

[56] References Cited

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4,928,065	5/1990	Lane et al.	324/464

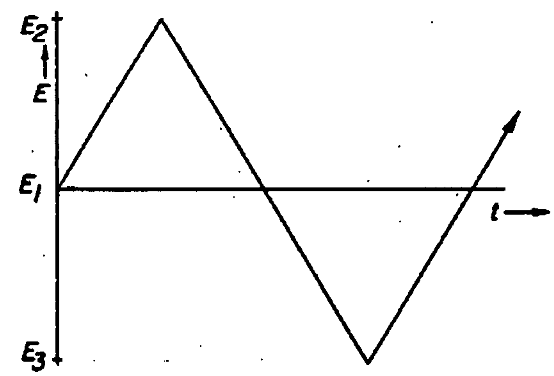
Primary Examiner—T. Tung

Attorney, Agent, or Firm—Killworth, Gottman, Hagan & Schaeff

[57] ABSTRACT

A method and apparatus is disclosed for the complete analysis of used oils, lubricants, and fluids. The method can monitor antioxidant depletion, oxidation initiators buildup, product buildup, or liquid contamination or combinations thereof. The method can be performed either on-line or off-line but is preferably an on-line system, either a built-in system or a dip-stick type system, having a working microelectrode, a reference electrode, and an auxiliary electrode. A sample is contacted by the electrodes and subjected to cyclic voltammetric analysis, whereby a varying electric current is produced within the sample. The current is measured and recorded, and the conductance is measured. The remaining useful life of the oil, lubricant, or fluid is then determined from the wave heights of the oxidation and reduction peaks, and the contamination is determined from the conductance.

19 Claims, 5 Drawing Sheets



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  - L5: (178481) l3 or l4
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  - L10: (35) l9 and l6
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DBs: USPAT; US-PGPUB

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	U	1	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C
1	☐	☐	☐	☐	US 6334945 B1	20020101	11	Aging process for solid electrode capacitor	205/687	205/229; 205/688;		Lessner, Philip Michael et al.	☑	☐

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6	US 5364522 A	9	☐	☑	☐	☐		USPAT
7	US 5147515 A	8	☐	☑	☐	☐		USPAT
8	US 5141602 A	27	☐	☑	☐	☐		USPAT
9	US 4879018 A	5	☐	☑	☐	☐		USPAT
10	US 4656411 A	18	☐	☑	☐	☐		USPAT

US-PAT-NO: 4656411

DOCUMENT-IDENTIFIER: US 4656411 A

TITLE: DC current controller for hydroset battery electrode electroformation

---------- KWIC ---------

Detailed Description Text - DETX (6):
Following the signal flow from the regulator block 23 in FIG. 1, to the next functional block of FIG. 1, that is, the PWM duty cycle generator 24 (FIG. 1). The common approach to build a duty cycle generator is to form a saw tooth wave (triangular) and vary a reference over the slope of the saw tooth wave form.

||

Detailed Description Text - DETX (9):
Considering the large and non-linear changes that can take place, a new approach had to be conceived that is cost effective and simple. The circuit that fits into the PWM duty cycle generator block 24 shown in FIG. 1 is detailed by Q1, Q2, Q3, and IC2 shown in FIG. 4 and their related components. This circuit has a fixed reference and the saw tooth varies in amplitude, but the saw tooth is actually trapezoidal with an adjustable slope. The slope is the important factor that makes the circuit respond to the large changes in load and circit impedences. FIGS. 8A-8D shows the wave form that is developed by the circuit in block 24 and has the variable slope adjustment capability.

Detailed Description Text - DETX (10):
VR is the reference voltage that is constant and set by the voltage divider resistors R14 and R15 and is one input to the non inverting input IC2. NPN transistor Q1, R8, R9, R10 and R11 form a linear amplifier with fast response from a signal input from IC1. PNP transistor Q2, R12, C4 and R13 form a controlled charge rate circuit to form the slope and sides of the trapezoid controlled by the input of Q1. If R13=0 OHMS the wave form would be saw tooth or triangular. Increasing the OHMIC value of R13 flattens the slope for a faster response. The combination of a fast Q1 amplifier to an IC1 input and the correct slope fixed by R13 makes the PWM regulate better than +/-2% on load current. IC2 (FIG. 4) is the voltage comparator that compares the reference VR to the crossing points on trapezoidal wave form as shown in FIGS. 8A-8D. IC2 finds the crossing points and outputs the square wave. NPN transistor Q3 is a reset switch controlled by signal pulses from the oscillator 26. The frequency of the oscillator 26 determines the switch rate period "T" and the input from IC1 determines the voltage height of the trapezoidal wave form of time on and time off of period "T". C5 is a positive feed back capacitor of several hundred picofarads that increases the slew rate of the operational amplifier IC2 that squares the output wave form.

Current US Cross Reference Classification - CCXR (1):
205/63

United States Patent [19]

Carlson

[11] Patent Number: 4,656,411

[45] Date of Patent: Apr. 7, 1987

[54] DC CURRENT CONTROLLER FOR HYDROSET BATTERY ELECTRODE ELECTROFORMATION

[76] Inventor: Daniel P. Carlson, 10034 Plum Rd., Erie, Pa. 16510

[21] Appl. No.: 671,113

[22] Filed: Nov. 13, 1984

[51] Int. Cl.⁴ H01M 10/44

[52] U.S. Cl. 320/16; 320/17; 320/19; 320/21; 320/40; 204/2.1

[58] Field of Search 320/15-19, 320/21, 39, 40; 363/41, 121, 124; 204/2.1, 6

[56] References Cited

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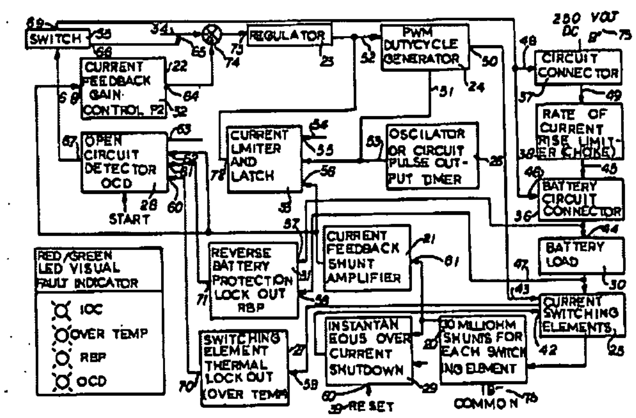
Primary Examiner—W. B. Perkey
Attorney, Agent, or Firm—Charles L. Lovercheck; Wayne L. Lovercheck; Dale R. Lovercheck

[57] ABSTRACT

A closed loop regulating system suitable for battery plate formation is provided. The circuit incorporates MOS FET and provides compensation for resistance changes that occur in MOS FET devices due to temperature changes. The regulating system also has means to precisely compensate for substantial impedance changes that occur over long time periods of an electroforming cycle for loads of various sizes.

37 Claims, 16 Drawing Figures



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  - L3: (3) (microarc or microarcs) near2 plasma
  - L4: (6316) l2 or l3
  - L5: (36) l4 and l1
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DBs: USPAT; US-PGPUB

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4	US 6368467 B1	7	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
5	US 6277253 B1	16	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
6	US 6183888 B1	9	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
7	US 5939215 A	5	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

US-PAT-NO: 6585875

DOCUMENT-IDENTIFIER: US 6585875 B1

TITLE: Process and apparatus for cleaning and/or coating metal surfaces using electro-plasma technology

---------- KWIC ---------

Brief Summary Text - BSTX (16):
WO-A-97/35052 describes an electrolytic process for cleaning electrically conducting surfaces using an electro-plasma (arc discharge) in which a liquid electrolyte flows through one or more holes in an anode held at a high DC voltage and impinges on the workpiece (the cathode) thus providing an electrically conductive path. The system is operated in a regime in which the electrical current decreases or remains substantially constant with increase in the voltage applied between the anode and the cathode and in a regime in which discrete bubbles of gas and/or vapour are present on the surface of the workpiece during treatment.

Brief Summary Text - BSTX (22):
We have now developed an improved process in which an electro-plasma (arc discharge) is employed to clean and/or to apply a metal coating to an electrically conductive surface, for example, steel, in which the electrically conductive pathway is provided by a foaming electrolyte which fills the space between the anode and the cathode and provides advantages with respect to lower power consumption, more uniform surface treatment and greater latitude in the size of the gap between anode and workpiece.

Current US Original Classification - CCOR (1):
205/87

Current US Cross Reference Classification - CCXR (10):
205/118

Current US Cross Reference Classification - CCXR (11):
205/138

Current US Cross Reference Classification - CCXR (12):
205/148

Current US Cross Reference Classification - CCXR (15):
205/88

Current US Cross Reference Classification - CCXR (16):
205/98

(12) **United States Patent**
Ryabkov

(10) Patent No.: **US 6,585,875 B1**
(45) Date of Patent: **Jul. 1, 2003**

(54) PROCESS AND APPARATUS FOR CLEANING AND/OR COATING METAL SURFACES USING ELECTRO-PLASMA TECHNOLOGY

(75) Inventor: Danila Vitalievich Ryabkov, Moscow (RU)

(73) Assignee: CAP Technologies, LLC, Baton Rouge, LA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 10/031,978

(22) PCT Filed: Jul. 28, 2000

(86) PCT No.: PCT/GB00/02917

§ 371 (c)(1), (2), (4) Date: Jan. 25, 2002

(87) PCT Pub. No.: WO01/09410

PCT Pub. Date: Feb. 8, 2001

(30) Foreign Application Priority Data

Jul. 30, 1999 (RU) 99116537

(51) Int. Cl.[7] C25D 5/00; C25F 1/00

(52) U.S. Cl. .... 205/87; 204/164; 204/206; 204/224 R; 204/232; 204/237; 204/239; 204/241; 204/269; 204/275.1; 205/88; 205/98; 205/118; 205/138; 205/148; 205/705; 205/722

(58) Field of Search .... 205/80, 87, 88, 205/98, 102, 118, 138, 148, 705, 710, 722; 204/164, 224 R, 232, 237, 239, 241, 202, 203, 206, 207, 269, 274, 275.1, 284

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International Preliminary Examination Report, International Application No. PCT/GB00/02917, filed Jul. 28, 2000, Applicant: Danila Vitalievich Ryabkov, no publication date.

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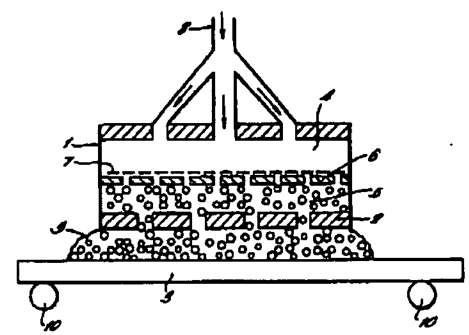
Primary Examiner—Roy King
Assistant Examiner—William T. Leader
(74) *Attorney, Agent, or Firm*—Brooks & Kushman P.C.

(57) ABSTRACT

A process for cleaning an electrically conducting surface (3) by arranging for the surface to form the cathode of an electrolytic cell in which the anode (1) is maintained at a DC voltage in excess of 30V and an electrical arc discharge (electro-plasma) is established at the surface of the workpiece by suitable adjustment of the operating parameters, characterized in that the working gap between the anode and the cathode is filled with an electrically conductive medium consisting of a foam (9) comprising a gas/vapor phase and a liquid phase. The process can be adapted for simultaneously coating the metal surface by including ions of the species required to form the coating in the electrically conductive medium. Apparatus for carrying out the process is also disclosed and, in particular, an anode assembly (1) which comprises a perforated anode plate (2) which is in communication with a chamber (4) adapted to receive a flow of a liquid electrolyte, means to supply the liquid electrolyte to the chamber, and means (7) to convert the liquid electrolyte received in the chamber into a foam.

18 Claims, 4 Drawing Sheets



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L3: (3) (microarc or microarcs) near2 plasma
L4: (6316) l2 or l3
L5: (36) l4 and l1
L6: (3949) ceramic adj coating
L7: (59) l1 and l6
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DBs: USPAT; US-PGPUB

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l1 and l6

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3	US 6562223 B2	14	☑	USPAT
4	US 6524718 B1	4	☑	USPAT
5	US 6475644 B1	26	☑	USPAT
6	US 6332937 B1	15	☑	USPAT
7	US 6290834 B1	5	☑	USPAT

Brief Summary Text - BSTX (8):
A desirable improved process would be one which has little or no ceramic re-melt or liquid flow on application of the laser to cut the ink-well patterns. Additionally, a desirable process would leave holes or cells having sharply defined patterns, having walls which are smooth and of the same texture as the surrounding area, to facilitate ink (and other material to be transferred) removal and cleanup as well as more precise patterns. Easier cleanup increases productivity and minimizes the chances of damage to the roll surface. And, a desirable process would be one which does not demonstrate a significant shortcoming of the thermal spray processes--the sometimes inadequate adherence of the coating to the roll substrate. This is an inherent difficulty with the thermal spray processes particularly for substrates, such as aluminum, having coefficients of thermal expansion considerably different from the ceramic coating.

Brief Summary Text - BSTX (9):
Further, it would be desirable to have a process wherein the laser-produced cell patterns are imparted to the roll surface prior to application of the ceramic coating rather than after, since application of the laser after coating incorporates all the above possible defects and shortcomings, and results in vertical surfaces in the cells which are different in composition from the horizontal surfaces. Coating after engraving is not feasible with sprayed ceramics.

Brief Summary Text - BSTX (16):
In another version of my invention, a hard wear resistant ceramic coating is formed on an incipient process roll having a blank metal surface by immersing it in an selectrolytic bath comprising (deionized) water, an electrolytic agent comprising an alkali metal salt or hydroxide (preferably potassium hydroxide) at a concentration of 0.5-7 grams per liter, and, as a passivating agent, a colloidal suspension of sodium silicate in the form Na.sub.2 O.xSiO.sub.2 (x=>2.55 by weight) at a concentration of 2.0-9.5 grams per liter while conducting through the bath a modified shaped-wave alternating electric current from a source of at least 250-800 volts through the surface of the incipient printing (process) roll. The modified shaped-wave electric current rises from zero to its maximum height and falls to below 40% of its maximum height within less than a quarter of a full alternating cycle, thereby causing dielectric breakdown.and the formation of a compact ceramic film on the roll surface. I then remove the roll from the bath, grind or polish the formed ceramic coating to a suitably smooth finish, and engrave or emboss its surface, preferably by laser, to impose a three-dimensional pattern on the ceramic coating surface of the roll.

Detailed Description Text - DETX (2):
The electrolytic treatment of the incipient liquid transfer roll will generally take about 30 to about 240 minutes to form a ceramic coating of 25 to 300 microns (0.001 to 0.012 inch) thick. A preferred thickness for the coating is 100 to 200 microns (0.004 to 0.012 inch) thick. Where the roll is aluminum, during the process cycle the substrate temperature is preferably maintained at less than 60.degree. C. (140.degree. F.). The incipient blank liquid transfer roll will preferably have an aluminum surface, but it may be made of aluminum, magnesium, titanium, zirconium, beryllium, hafnium or alloys thereof.

Detailed Description Text - DETX (7):

(12) **United States Patent**
Pearsall

(10) Patent No.: **US 6,290,834 B1**
(45) Date of Patent: **Sep. 18, 2001**

(54) **CERAMIC COATED LIQUID TRANSFER ROLLS AND METHODS OF MAKING THEM**

(75) Inventor: Thomas J. Pearsall, Summerfield, FL (US)

(73) Assignee: Ceramic Coatings Technologies, Inc., Palm City, FL (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/548,095

(22) Filed: Apr. 12, 2000

(51) Int. Cl.[7] C25D 5/34
(52) U.S. Cl. 205/205; 205/120; 205/220
(58) Field of Search 205/220, 102, 205/205

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5,857,950 1/1999 Hycner 492/30

Primary Examiner—Kathryn Gorgos
Assistant Examiner—Thomas H Parsons
(74) Attorney, Agent, or Firm—William L. Krayer

(57) ABSTRACT

Liquid transfer rolls such as printing rolls are made by imposing a hard ceramic coating on an incipient liquid transfer roll in an electrolytic bath subjected to a modified shaped wave alternating current, which causes dielectric breakdown and the formation of a hard ceramic coating on the roll. The roll is laser engraved to form liquid carrying reservoirs or cells on the surface of the roll, either before or after the ceramic coating is built on the roll.

19 Claims, No Drawings

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5	US 6475644 B1	26		☑				USPAT
6	US 6332937 B1	15		☑				USPAT
7	US 6290834 B1	5		☑				USPAT
8	US 6261422 B1	9		☑				USPAT
9	US 6254759 B1	13		☑				USPAT
10	US 6248227 B1	8		☑				USPAT
11	US 6197178 B1	10		☑				USPAT

US-PAT-NO: 6197178

DOCUMENT-IDENTIFIER: US 6197178 B1

TITLE: Method for forming ceramic coatings by micro-arc oxidation of reactive metals

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Brief Summary Text - BSTX (5):

The practice of coating a metal substrate with a thin layer of a ceramic material has been used commercially for many years. One purpose of providing a ceramic coating of a metal is to improve the wear resistance of the metal from abrasion and another purpose is to protect the surface of the metal from thermal degradation, oxidation, or corrosion. In particular, research and development activity has been carried out throughout the world to provide superior surface properties to metals such as aluminum and aluminum alloys. Aluminum and aluminum alloys are extremely desirable metals for manufacturing machinery components and the like because they are relatively inexpensive and have relatively low densities. Aluminum and aluminum alloys, however, have the drawbacks of being relatively soft and do not resist wear and abrasion very well. In addition, aluminum is chemically active so that it tends to react with chemicals and even moisture in the air, thereby corroding.

Brief Summary Text - BSTX (6):

A known method of improving the surface of a substrate of aluminum or an alloy of aluminum is to apply a ceramic coating to the substrate by spraying the ceramic coating onto the substrate. Typically, the process of "flame spraying" includes a wire-type flame sprayer. The protective coatings applied in this manner are limited to those materials which can be formed into a wire or rod.

Brief Summary Text - BSTX (9):

Ceramic coatings used in the prior art are generally inherently porous and ordinarily do not provide much oxidation or corrosion protection to the base material. Thus, undercoats made from oxidation-resistant materials, or alloys are used between the base material and the ceramic coating if the substrate material is not corrosion resistant.

Brief Summary Text - BSTX (11):

Typically, one class of ceramic coatings has high thermal resistance and a low wear resistance, while another class of ceramic coatings has a high wear resistance and has a low thermal resistance. The general reason for this relationship is that ceramic coatings which have a high thermal resistance typically are more porous and have a higher void content thereby providing a good thermal barrier but also being less resistant to abrasion. A ceramic coating having a high abrasion resistance has a low void content, thus reducing damage to abrasion. Furthermore, in the prior art, only one specimen is connected to one electrode and the other electrode is connected to the electrolyte tank. Primarily, the power source was single phase AC and DC power.

(12) **United States Patent** (10) Patent No.: **US 6,197,178 B1**

Patel et al. (45) Date of Patent: **Mar. 6, 2001**

(54) **METHOD FOR FORMING CERAMIC COATINGS BY MICRO-ARC OXIDATION OF REACTIVE METALS**

(75) Inventors: Jerry L. Patel, Peabody; Nannaji Saka, Cambridge, both of MA (US)

(73) Assignee: Microplasmic Corporation

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/285,604

(22) Filed: Apr. 2, 1999

(51) Int. Cl.^{7} C25D 11/02; C25D 21/12

(52) U.S. Cl. 205/81; 204/230.2; 205/96; 205/107; 205/137; 205/145; 205/321; 205/322; 205/324; 205/325; 205/326

(58) Field of Search 205/81, 82, 83, 205/96, 107, 137, 145, 320, 321, 322, 324, 325, 326, 328; 204/229.8, 230.2

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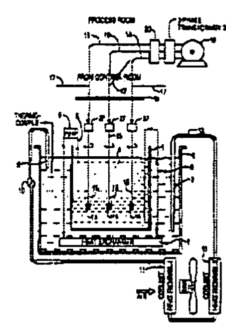
Primary Examiner—Kathryn Gorgos
Assistant Examiner—William T. Leader

(57) **ABSTRACT**

A process and apparatus for forming oxide coatings on bodies of aluminum and aluminum alloys are described. The process includes forming an electrolyte bath in an inert container. At least two reactive metal bodies are suspended in the bath. The bodies are connected to electrodes which, in turn, are connected to a multiphase AC circuit. A multiphase power (preferably three-phase between three bodies) potential is imposed between each of the bodies. The bodies are moved in the electrolyte bath relative to each other until micro-arcs occur on the surfaces of the bodies, whereby to commence oxidation of the bodies. The imposition of the potential between each of the bodies is continued until the desired thickness of oxide is formed on the bodies.

30 Claims, 2 Drawing Sheets



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25	US 5344551 A	17	☐	☑	☐	☐		USPAT
26	US 5296260 A	19	☐	☑	☐	☐		USPAT
27	US 5281326 A	3	☐	☑	☐	☐		USPAT
28	US 5217597 A	6	☐	☑	☐	☐		USPAT
29	US 5205921 A	4	☐	☑	☐	☐		USPAT
30	US 5147515 A	8	☐	☑	☐	☐		USPAT

US-PAT-NO: 5147515

DOCUMENT-IDENTIFIER: US 5147515 A

TITLE: Method for forming ceramic films by anode-spark discharge

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Brief Summary Text - BSTX (35):
Low outgassing properties, corrosion resistance and fastness properties can be imparted to an apparatus for manufacturing semiconductor devices by applying a ceramic film onto the shroud or the chamber of a reaction vessel of the apparatus according to the method of this invention. Moreover, if an aluminum or aluminum clad copper conductors is provided with a ceramic coating, there can be obtained an electric wire coated with the ceramic layer having high dielectric breakdown voltage in addition to high flexibility and whose coated layer is hardly broken even if the layer has a flaw.

Current US Cross Reference Classification - CCXR (1):
205/320

Current US Cross Reference Classification - CCXR (2):
205/321

Current US Cross Reference Classification - CCXR (3):
205/322

Current US Cross Reference Classification - CCXR (4):
205/323

US005147515A

United States Patent [19]

Hanagata et al.

[11] Patent Number: 5,147,515

[45] Date of Patent: Sep. 15, 1992

[54] METHOD FOR FORMING CERAMIC FILMS BY ANODE-SPARK DISCHARGE

[75] Inventors: Haruo Hanagata, Ebina; Tsukasa Suzuki; Kazuo Yanagida, both of Kasukabe; Hidesato Igarashi, Tokyo, all of Japan

[73] Assignee: Dipsol Chemicals Co., Ltd., Tokyo, Japan

[21] Appl. No.: 573,703

[22] Filed: Aug. 28, 1990

[30] Foreign Application Priority Data

Sep. 4, 1989 [JP] Japan 1-228639
Mar. 6, 1990 [JP] Japan 2-54827

[51] Int. Cl.[5] H05F 3/04

[52] U.S. Cl. 204/164; 205/320; 205/321; 205/322; 205/323; 427/37

[58] Field of Search 204/56.1, 58, 58.4, 204/164; 427/37; 106/628, 635, 637; 264/22; 205/321, 322, 323, 320

[56] References Cited

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Primary Examiner—John Niebling
Assistant Examiner—Brian M. Bolam
Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt

[57] ABSTRACT

A method for forming a ceramics film on the surface of a substrate comprises performing spark discharge in an electrolytic bath, wherein the electrolytic bath comprises an aqueous solution of a water-soluble or colloidal silicate and/or an oxyacid salt to which ceramics fine particles and/or specific fine particles are dispersed and the spark discharge is carried out in the electrolytic bath while ensuring the suspended state of the ceramics particles and/or the specific fine particles in the electrolytic bath. The method makes it possible to effectively form, on the surface of a metal substrate, ceramics films having a variety of color tones as well as excellent insulating properties and hardness. Moreover, it further makes it possible to effectively form a composite ceramics film having excellent wear resistance on the surface of a metal substrate.

13 Claims, No Drawings

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25	US 5344551 A	17	☐	☑	☐	☐		USPAT
26	US 5296260 A	19	☐	☑	☐	☐		USPAT
27	US 5281326 A	3	☐	☑	☐	☐		USPAT
28	US 5217597 A	6	☐	☑	☐	☐		USPAT
29	US 5205921 A	4	☐	☑	☐	☐		USPAT
30	US 5147515 A	8	☐	☑	☐	☐		USPAT

US-PAT-NO: 5147515

DOCUMENT-IDENTIFIER: US 5147515 A

TITLE: Method for forming ceramic films by anode-spark discharge

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Brief Summary Text - BSTX (35):

Low outgassing properties, corrosion resistance and fastness properties can be imparted to an apparatus for manufacturing semiconductor devices by applying a ceramic film onto the shroud or the chamber of a reaction vessel of the apparatus according to the method of this invention. Moreover, if an aluminum or aluminum clad copper conductors is provided with a ceramic coating, there can be obtained an electric wire coated with the ceramic layer having high dielectric breakdown voltage in addition to high flexibility and whose coated layer is hardly broken even if the layer has a flaw.

Current US Cross Reference Classification - CCXR (1):
205/320

Current US Cross Reference Classification - CCXR (2):
205/321

Current US Cross Reference Classification - CCXR (3):
205/322

Current US Cross Reference Classification - CCXR (4):
205/323

5,147,515

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solution. Specific examples thereof include oxide type ceramic such as Al_2O_3, $Al(OH)_3$, SiO_2, $3Al_2O_3.2SiO_2$, TiO_2, ZrO_2 and Cr_2O_3 and non-oxide type ceramics such as SiC, TiC, TiN, TiB, ZrB, BN, WC, WSi_2 and $MoSi_2$. These ceramic particles may be used alone or in combination.

The particle size of the ceramic particles desirably ranges from 0.03 to 100 μm, in particular 0.03 to 20 μm. That is, when the particle size thereof is increased, it is difficult to co-deposite the ceramic particles and if they are co-deposited the resulting film is non-uniform.

The amount of the fine particles of ceramic to be added to the electrolytic bath can be arbitrarily determined depending on the kinds of the electrolytes in which the fine particles are dispersed and the amount of the fine particles to be dispersed, but is in general up to 200 g/l and most preferably ranges from 5 to 100 g/l from the viewpoint of the efficiency of the deposition.

Examples of the fine particles used in the second aspect of the present invention are molybdenum disulfide, carbon, fluorinated graphite, tetrafluoroethylene resin or mixture thereof. Graphite is preferable as a carbon component used herein. These fine particles have self-lubricating properties, are hence taken in the ceramic film during the spark discharge to thus give a film having good wear resistance.

In this embodiment, the fine ceramic particles used in the first aspect of the invention can be used together with the fine particles having self-lubricating properties.

The particle size of the fine particles having self-lubricating properties desirably ranges from 0.01 to 100 μm and preferably 0.03 to 20 μm. That is, when the particle size thereof is increased, it is difficult to co-deposite the ceramic particles and if they are co-deposited the resulting film is non-uniform.

The amount of the fine particles having self-lubricating properties to be added to the electrolytic bath can be arbitrarily determined depending on the kinds of the electrolytes in which the fine particles are dispersed and the amount of the fine particles to be dispersed, but is in general up to 200 g/l and most preferably ranges from 5 to 100 g/l from the viewpoint of the efficiency of the deposition.

In the first and second aspects of the present invention, examples of the metal substrates on which a ceramic film can be formed by the spark discharge technique include those made from aluminum and alloys thereof; zirconium, titanium, niobium, magnesium and alloys thereof.

When a film is formed on a metal substate by spark discharge, the substrate must not be subjected to a particular pretreatment, but it is desirable to sufficiently clean the surface of the substrate through degreasing, etching, washing with an acid or the like.

An insoluble electrode is used as a cathode and the cathode may be formed from, for instance, iron, stainless steel, nickel or the like.

In the method of the present invention, the spark discharge is carried out in the electrolytic bath defined above while ensuring the suspended state of the ceramic particles in the electrolytic bath. The ceramic fine particles sediment due to the gravitational action or the self-weight and thus it is important to conduct the spark discharge while maintaining the suspended state of the particles in the usual manner. The retention of such suspended state can be performed by stirring or circulation of the electrolyte.

4

When fine particles having poor dispersion properties are employed, there may be used a dispersant, for instance, a surfactant such as cationic, non-ionic or anionic ones for obtaining a good dispersion.

The temperature of the electrolytic bath during the spark discharge in general ranges from 5° to 90° C. and preferably 15° to 60° C. This is because, if it is too low, the film-forming velocity by the spark discharge is low, while if it is too high, it is liable to form a non-uniform film.

In addition, if the current density used is too low, the fine particles are hardly deposited, while if it is too high, a film having a low particle density or a coarse film is formed at high current portions. Therefore, the current density preferably ranges from 0.2 to 20 A/dm², more preferably 1 to 5 A/dm².

The output from a power supply may be a direct current having any wave form, but preferably those having pulse shape (rectangular wave form), saw-tooth-wave form or DC half-wave form.

The spark discharge-initiating voltage varies depending on various factors such as the wave form of the output current from the dc power supply, the concentration of the silicate and that of the oxyacid salt and the temperature of the bath, but it desirably ranges from 50 to 200 V. Moreover, the voltage observed during the film formation is increased as the spark discharge proceeds and the final voltage sometimes exceeds 1,000 V.

The electrolysis time varies depending on the desired thickness of the resulting film. However, if the resulting film is thin, the film does not show the quality peculiar thereto. Therefore, the electrolysis must be performed for at least 5 minutes. In general, practically acceptable films having a thickness, for instance, ranging from 2 to 80 μm can be obtained if the electrolysis is performed for 10 to 60 minutes.

According to the first aspect of the present invention, there can effectively be prepared metallic materials having ceramic films having high insulating properties, high hardness and a variety of color tones.

Low outgassing properties, corrosion resistance and fastness properties can be imparted to an apparatus for manufacturing semiconductor devices by applying a ceramic film onto the shroud or the chamber of a reaction vessel of the apparatus according to the method of this invention. Moreover, if an aluminum or aluminum clad copper conductors is provided with a ceramic coating, there can be obtained an electric wire coated with the ceramic layer having high dielectric breakdown voltage in addition to high flexibility and whose coated layer is hardly broken even if the layer has a flaw.

According to the method of this invention, the color tone of the resulting films is rather white depending on the kinds of the fine particles used and, therefore, the method can also be useful as a whitening treatment for aluminum construction materials.

If a ceramic film is applied onto a container for cosmetics comprising an aluminum material according to the method of this invention, there can be obtained a container for cosmetics having beautiful appearance of a variety of color tones and free of hit marks.

In addition, if a ceramic film is applied onto a heater of aluminum, a far infrared radiator having excellent far infrared emission properties and free of hit marks can be obtained.

The second aspect of the present invention makes it possible to effectively produce metallic materials hav-

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  - L4: (6316) 12 or 13
  - L5: (36) 14 and 11
  - L6: (3949) ceramic adj coating
  - L7: (59) 11 and 16
  - L8: (15154) arc same discharge
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11 and 18

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	Document ID	Pages						U	S	C	P	Kind Codes	Source
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7	US 6325909 B1	15	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
8	US 6277253 B1	16	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
9	US 5879532 A	14	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
10	US 5827420 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
11	US 5723038 A	4	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
12	US 5385662 A	4	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5385662

DOCUMENT-IDENTIFIER: US 5385662 A

TITLE: Method of producing oxide ceramic layers on barrier layer-forming metals and articles produced by the method

---------- KWIC ---------

Brief Summary Text - BSTX (6):
Because of the pattern of the current density/potential curves for the anodic spark discharge, three distinct portions can be distinguished. i.e. the Faraday portion, the spark discharge portion and the arc discharge portion, see P. Kurze mentioned above.

Current US Original Classification - CCOR (1):
205/316

Current US Cross Reference Classification - CCXR (1):
205/321

Current US Cross Reference Classification - CCXR (2):
205/322

Current US Cross Reference Classification - CCXR (3):
205/323

Current US Cross Reference Classification - CCXR (4):
205/324

United States Patent [19]

Kurze et al.

[11] Patent Number: 5,385,662

[45] Date of Patent: Jan. 31, 1995

[54] METHOD OF PRODUCING OXIDE CERAMIC LAYERS ON BARRIER LAYER-FORMING METALS AND ARTICLES PRODUCED BY THE METHOD

[75] Inventors: Peter Kurze, Düren; Dora Banerjee, Kerpen; Hans-Jürgen Kletke, Düren, all of Germany

[73] Assignee: Electro Chemical Engineering GmbH, Zug, Switzerland

[21] Appl. No.: 982,092

[22] Filed: Nov. 25, 1992

[30] Foreign Application Priority Data

Nov. 27, 1991 [DE] Germany 4139006

[51] Int. Cl.[6] C25D 9/06

[52] U.S. Cl. 205/316; 205/321; 205/322; 205/323; 205/324

[58] Field of Search 205/324, 329, 316, 333, 205/321, 322; 204/178

[56] References Cited

U.S. PATENT DOCUMENTS

3,862,892 1/1975 Lautenschlager et al. 205/324
4,481,083 11/1984 Ball et al. 205/324
4,869,789 9/1989 Kurze et al. 205/324
4,898,651 2/1990 Mahmood 205/329

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289065 4/1991 German Dem. Rep. .

OTHER PUBLICATIONS

Van et al. "Mechanism of Anodic Spark Deposition" in American Ceramic Society Bulletin (Jun., 1977) vol. 56, No. 6, pp. 563-566.

Primary Examiner—John Niebling
Assistant Examiner—Brendan Mee
Attorney, Agent, or Firm—Cohen, Pontani, Lieberman & Pavane

[57] ABSTRACT

A method of producing oxide ceramic layers on Al, Mg, Ti, Ta, Zr, Nb, Hf, Sb, W, Mo, V, Bi or their alloys by a plasma-chemical anodical oxidation in a chloride-free electrolytic bath having a pH value of 2 to 8 and a constant bath temperature of −30° to +15° C. A current density of at least 1 A/dm² is maintained constant in the electrolytic bath until the voltage reaches a predetermined end value.

7 Claims, No Drawings

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12	US 5385662 A	4	☐	☑	☐	☐		USPAT
13	US 5344551 A	17	☐	☑	☐	☐		USPAT
14	US 5290592 A	20	☐	☑	☐	☐		USPAT
15	US 5290424 A	14	☐	☑	☐	☐		USPAT
16	US 5268045 A	15	☐	☑	☐	☐		USPAT
17	US 5225069 A	7	☐	☑	☐	☐		USPAT

US-PAT-NO: 5225069

DOCUMENT-IDENTIFIER: US 5225069 A

TITLE: Process for the production of oxide ceramic surface films on silicon-containing light metal cast alloys

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Brief Summary Text - BSTX (5):
It is known to produce high-adhesion, dense and thick dispersion films on metals, especially on iron and iron work materials, by means of anodic arc-discharge or conventional thermal treatment by deposition from dispersion systems (DD-PS 151330).

Current US Original Classification - CCOR (1):
205/325

Current US Cross Reference Classification - CCXR (1):
205/332

US005225069A

United States Patent [19]

Haupt et al.

[11] Patent Number: 5,225,069

[45] Date of Patent: Jul. 6, 1993

[54] PROCESS FOR THE PRODUCTION OF OXIDE CERAMIC SURFACE FILMS ON SILICON-CONTAINING LIGHT METAL CAST ALLOYS

[75] Inventors: Kerstin Haupt; Jurgen Schmidt; Ulrich Bayer; Thomas Furche, all of Jena, Fed. Rep. of Germany

[73] Assignee: Jenoptik GmbH, Jena, Fed. Rep. of Germany

[21] Appl. No.: 884,691

[22] Filed: May 18, 1992

[30] Foreign Application Priority Data

May 21, 1991 [DE] Fed. Rep. of Germany 4116910

[51] Int. Cl.5 .... C25D 11/16

[52] U.S. Cl. 205/325; 205/332

[58] Field of Search 205/325, 332

[56] References Cited

FOREIGN PATENT DOCUMENTS

28399 2/1991 Japan .

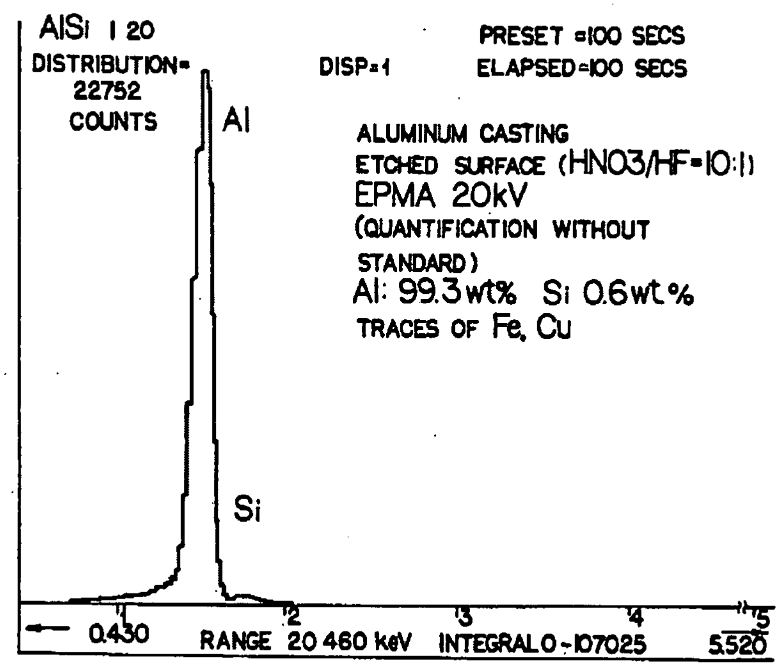
Primary Examiner—T. M. Tufariello

Attorney, Agent, or Firm—McAulay Fisher Nissen Goldberg & Kiel

[57] ABSTRACT

A process for the production of white and black oxide ceramic surface films on silicon-containing light metal cast alloys by plasma-chemical anodic oxidation. An aluminum cast alloy is pickled with nitric acid and hydrofluoric acid and coated by plasma-chemical anodic oxidation in an aqueous electrolyte. Accordingly, a coating variant is provided particularly for construction parts of silicon-containing light metal cast alloys having complicated shapes which enables the production of uniformly thin oxide ceramic surface films in contrast to conventional coating variants.

8 Claims, 2 Drawing Sheets



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cl 5 17 pulsed current 200-1000 Hz

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16	US 5268045 A	15	☐	☑	☐	☐		USPAT
17	US 5225069 A	7	☐	☑	☐	☐		USPAT
18	US 5085742 A	5	☐	☑	☐	☐		USPAT
19	US 4950365 A	6	☐	☑	☐	☐		USPAT
20	US 4869789 A	3	☐	☑	☐	☐		USPAT

US-PAT-NO: 4869789

DOCUMENT-IDENTIFIER: US 4869789 A

TITLE: Method for the preparation of decorative coating on metals

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Brief Summary Text - BSTX (15):
It has been found that voltages which are associated with the arc-discharge region in the current-voltage characteristic of the electrolyte-metal pairing, and which usually cause destruction of the coating, do yield an all-round homogeneous coating in connection with pulse operation up to 300 Hz.

Current US Original Classification - CCOR (1):
205/108

Current US Cross Reference Classification - CCXR (1):
205/322

Current US Cross Reference Classification - CCXR (2):
205/324

Claim 5 pulse 35-300Hz

United States Patent [19]

Kurze et al.

[11] Patent Number: 4,869,789

[45] Date of Patent: Sep. 26, 1989

[54] METHOD FOR THE PREPARATION OF DECORATIVE COATING ON METALS

[75] Inventors: Peter Kurze, Oberlichtenau; Waldemar Krysmann; Maria Berger, both of Karl-Marx-Stadt; Klaus Rabending, Taura; Joachim Schreckenbach, Limbach-Oberfrohna; Thomas Schwarz, Karl-Marx-Stadt; Karl-Heinz Hartmann, Güntersberge, all of German Democratic Rep.

[73] Assignee: Technische Universitaet Karl-Marx-Stadt, Karl-Marx-Stadt, German Democratic Rep.

[21] Appl. No.: 151,363

[22] Filed: Feb. 2, 1988

[30] Foreign Application Priority Data

Feb. 2, 1987 [DD] German Democratic Rep. ... 299618
Feb. 2, 1987 [DD] German Democratic Rep. ... 229620

[51] Int. Cl.[4] C25D 11/02; C25D 11/04

[52] U.S. Cl. 204/56.1; 204/58

[58] Field of Search 204/56.1, 58

[56] References Cited

U.S. PATENT DOCUMENTS

4,144,142 3/1979 Takahashi 204/58

Primary Examiner—T. M. Tufariello
Attorney, Agent, or Firm—Jordan and Hamburg

[57] ABSTRACT

The process for producing decorative coatings on metals is primarily used in the jewelry industry, in handicrafts, and in device construction according to the invention, matte, true-to-form decorative coatings with a homogenous thickness of 3 μm to 30 μm are provided means of an electrochemical and plasmachemical reaction using a pulse operation in aqueous electrolytes at voltage peaks of 250 V to 750 V, pulse times of 20 μs to 2 ms, pulse frequencies of 35 Hz to 300 Hz, pulse currents from 20 A to 120 A, electrolyte temperatures between 318 K and 360 K, and average current densities from 0.1 A cm^{-2} to 1 A cm^{-2}.

9 Claims, No Drawings

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  - L4: (6316) l2 or l3
  - L5: (36) l4 and l1
  - L6: (3949) ceramic adj coating
  - L7: (59) l1 and l6
  - L8: (15154) arc same discharge
  - L9: (32) l1 and l8
  - L12: (3205) spark adj discharge
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2	US 6487969 B2	6	☐	☑	☐	☐		USPAT
3	US 6368467 B1	7	☐	☑	☐	☐		USPAT
4	US 6365028 B1	6	☐	☑	☐	☐		USPAT
5	US 6358391 B1	7	☑	☐	☐	☐		USPAT
6	US 5723038 A	4	☑	☐	☐	☐		USPAT
7	US 5720866 A	9	☑	☐	☐	☐		USPAT

US-PAT-NO: 6365028

DOCUMENT-IDENTIFIER: US 6365028 B1

TITLE: Method for producing hard protection coatings on articles made of aluminum alloys

---------- KWIC ---------

Brief Summary Text - BSTX (9):

A method is known for applying solid corrosion-resistant coatings to items made of aluminium and its alloys (U.S. Pat. No. 5,275,713) in an aqueous electrolyte solution containing an alkaline metal silicate, hydrogen peroxide and small quantities of hydrogen fluoride, alkaline metal hydroxide and a metal oxide (for example, molybdenum oxide). The solution has a pH of 11.2-11.8. A positive potential is delivered to the item from a direct or pulsed current source. For the first 1-60 s the voltage is raised to 240-260 V, and over the next 1-20 minutes (depending on the required coating thickness) it is steadily increased to 380-420 V. The introduction of hydrogen peroxide as an oxygen accumulator into the electrolyte helps to raise the rate of increase of the oxide coating and its hardness through intensification of oxide coating of the metal in the spark discharge zone.

Brief Summary Text - BSTX (17):

The oxide coating time may not be shortened by raising the electrical parameters of the electrolysis, for example the current density (above 30 A/dm.sup.2), because of a deterioration in the quality of the coating and a steep rise in the energy consumption of the process. The time of transition from the anodising stage to the spark discharge stage, however, depends on the initial current density.

Current US Original Classification - CCOR (1):
205/102

Current US Cross Reference Classification - CCXR (1):
205/103

Current US Cross Reference Classification - CCXR (2):
205/106

Current US Cross Reference Classification - CCXR (3):
205/323

Current US Cross Reference Classification - CCXR (4):
205/326

(12) **United States Patent**
Shatrov

(10) Patent No.: **US 6,365,028 B1**
(45) Date of Patent: **Apr. 2, 2002**

(54) METHOD FOR PRODUCING HARD PROTECTION COATINGS ON ARTICLES MADE OF ALUMINUM ALLOYS

(75) Inventor: Alexandr Sergeevich Shatrov, Moscow (RU)

(73) Assignee: Isle Coat Limited (GB)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/581,494

(22) PCT Filed: Dec. 17, 1997

(86) PCT No.: PCT/RU97/00408

§ 371 Date: Jun. 14, 2000

§ 102(e) Date: Jun. 14, 2000

(87) PCT Pub. No.: WO99/31303

PCT Pub. Date: Jun. 24, 1999

(51) Int. Cl.⁷ C25D 5/18
(52) U.S. Cl. 205/102; 205/103; 205/106; 205/323; 205/326
(58) Field of Search 205/102, 106, 205/107, 108, 103, 323, 324, 325, 326; 204/DIG. 9

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RU	94023296	10/1996
RU	2070622	12/1996
RU	2070947	12/1996
WO	WO9518250	7/1995

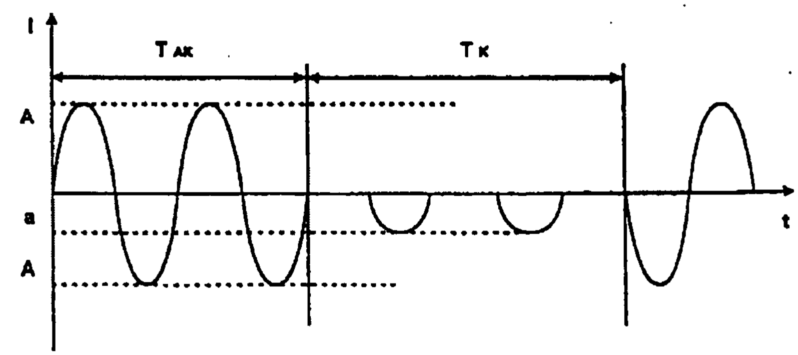
* cited by examiner

Primary Examiner—Kathryn Gorgos
Assistant Examiner—Wesley A. Nicolas
(74) *Attorney, Agent, or Firm*—Sheridan Ross P.C.

(57) ABSTRACT

This invention relates to the sphere of plasma electrolyte oxide coating of aluminium alloys. The method incorporates anode-cathode oxide coating in an alkaline electrolyte at a temperature of 15–50° C., using 50–60 Hz frequency alternating current. In the initial stage of the process oxide coating is carried on for 5–90 seconds at a current density of 160–180 A/dm², then the current density is dropped to 3–30 A/dm² and the process is continued in a regimen of spontaneous diminution of power demand without on-line adjustment of the regimen until the set coating thickness is achieved. The alkaline electrolyte used is an aqueous solution of alkaline metal hydroxide at 1–5 g/l, an alkaline metal silicate at 2–15 g/l, an alkaline metal pyrophosphate at 2–20 g/l and peroxide compounds at 2–7 g/l (in terms of H_2O_2—30%). The method enables the protective properties of ceramic oxide coatings to be enhanced through an increase in the micro-hardness, density and strength of adhesion to the substrate without any additional energy outlay or time required.

4 Claims, 1 Drawing Sheet



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2	US 5981084 A	13	☐	☑	USPAT
3	US 5958604 A	17	☐	☑	USPAT
4	US 5700366 A	12	☑	☐	USPAT
5	US 20030085113 A	15	☑	☐	US-PGPUB

US-PAT-NO: 5958604

DOCUMENT-IDENTIFIER: US 5958604 A
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TITLE: Electrolytic process for cleaning and coating electrically conducting surfaces and product thereof

---------- KWIC ---------

INVENTOR - INNM (2):
Steblianko; Valerij L.

United States Patent [19]

Riabkov et al.

[11] **Patent Number: 5,958,604**

[45] **Date of Patent: *Sep. 28, 1999**

[54] **ELECTROLYTIC PROCESS FOR CLEANING AND COATING ELECTRICALLY CONDUCTING SURFACES AND PRODUCT THEREOF**

[75] Inventors: **Vitalij M. Riabkov**, Moscow; **Valerij L. Steblianko**, Magnitogorsk, both of Russian Federation

[73] Assignee: **Metal Technology, Inc.**, Mandeville, La.

[*] Notice: This patent is subject to a terminal disclaimer.

[21] Appl. No.: **08/934,553**

[22] Filed: **Sep. 22, 1997**

Related U.S. Application Data

[63] Continuation-in-part of application No. 08/706,914, Sep. 3, 1996, Pat. No. 5,700,366.

[30] **Foreign Application Priority Data**

Mar. 20, 1996 [RU] Russian Federation 96104583

[51] **Int. Cl.**[6] **C25D 5/08**

[52] **U.S. Cl.** 428/612; 205/87; 205/95; 205/102; 205/131; 205/148; 205/151; 205/219; 428/687; 428/935

[58] **Field of Search** 205/87, 95, 102, 205/131, 148, 151, 219, 705, 712, 714, 715, 716; 428/612, 687, 935

[56] **References Cited**

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08003794	1/1996	Japan .
1244216	7/1986	U.S.S.R. .
1599446	10/1990	U.S.S.R. .
1306337	2/1973	United Kingdom .
1399710	2/1975	United Kingdom .
1436744	5/1976	United Kingdom .

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A.V. Timoshenko et al., "The Effect of Silicates in Sodium–Hydroxide Solution . . . by Microarc Oxidation" in *Protection of Metals*, vol. 30, No. 2, 1944, pp. 175–180. No Month Available.

Primary Examiner—Kathryn Gorgos
Assistant Examiner—William T. Leader
Attorney, Agent, or Firm—Watson Cole Grindle Watson, P.L.L.C.

[57] **ABSTRACT**

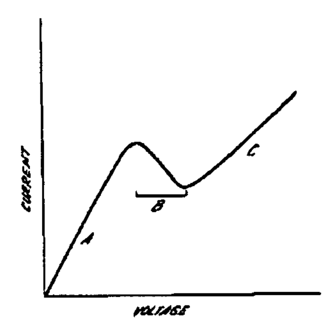
An electrolytic process for metal-coating the pre-cleaned surface of a workpiece of an electrically conducting material, which process comprises:

i) providing an electrolytic cell with a cathode comprising the workpiece and an anode comprising the metal for metal-coating of the surface of the workpiece;

ii) introducing an electrolyte into the zone created between the anode and the cathode by causing it to flow under pressure through at least one opening in the anode impinge on the cathode; and

iii) applying a voltage between the anode and the cathode and operating in a regime in which the electrical current decreases or remains substantially constant with increase in the voltage applied between the anode and the cathode, and in a regime in which discrete gas bubbles are present on the surface of the workpiece during treatment.

25 Claims, 7 Drawing Sheets



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TITLE: Electrolytic process for cleaning and coating electrically conducting surfaces

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United States Patent [19]

Steblianko et al.

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[45] **Date of Patent: Dec. 23, 1997**

[54] **ELECTROLYTIC PROCESS FOR CLEANING AND COATING ELECTRICALLY CONDUCTING SURFACES**

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[73] Assignee: **Metal Technology, Inc.**, Mandeville, La.

[21] Appl. No.: **706,914**

[22] Filed: **Sep. 3, 1996**

[30] **Foreign Application Priority Data**

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[51] Int. Cl.[6] C25D 5/08

[52] U.S. Cl. 205/87; 205/102; 205/131; 205/148; 205/219

[58] Field of Search 205/87, 95, 131, 205/148, 151, 219, 705, 714, 715, 716

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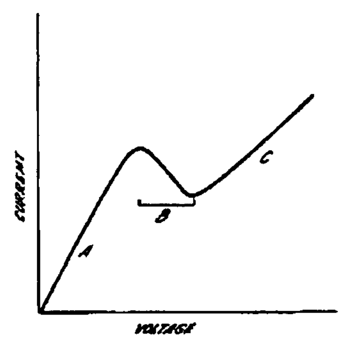
[57] **ABSTRACT**

An electrolytic process for simultaneously cleaning and metal-coating the surface of a workpiece of an electrically conducting material, which process comprises: i) providing an electrolytic cell with a cathode comprising the surface of the workpiece and an anode comprising the metal for metal-coating of the surface of the workpiece; ii) introducing an electrolyte into the zone created between the anode and the cathode by causing it to flow under pressure through at least one opening in the anode and thereby impinge on the cathode; and iii) applying a voltage between the anode and the cathode and operating in a regime in which the electrical current decreases or remains substantially constant with increase in the voltage applied between the anode and the cathode, and in a regime in which discrete gas bubbles are present on the surface of the workpiece during treatment.

23 Claims, 4 Drawing Sheets



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